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Production and characterization of co-composted biochar and digestate from biomass anaerobic digestion

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Abstract



Biochar, produced through pyrolysis of lignocellulosic biomass, is attracting increasing interest as soil amendment thanks to its potential numerous benefits to agriculture, as well as its ability to sequester carbon in soil. Solid fraction of digestate from anaerobic digestion is a well-known N-rich substrate, most often composted in large and small agro-industrial plants. Co-composting biochar and digestate has the potential to synergistically increase the agronomic value of both components: however, it needs further process and on-field research. The present research work reports on the experimental tests on producing biochar and co-composting various biochar amounts with digestate from biomass anaerobic digestion (product here named COMBI). Biochar was produced by feeding wood chips from chestnut to an innovative oxidative reactor. In order to evaluate the quality of the products obtained by composting and co-composting, correlating this with the final biochar rate in the material, the net organic matter yield, the humified organic matter, the compliance with the European Compost Network Quality Assurance Scheme (ECN-QAS) limits for inorganic pollutants, and the product stabilization and sanitization indexes were investigated. The 11.2% w/w d.b. biochar rate in the initial blend (19.8% w/w d.b final concentration in the co-composting were also assessed, as the reduced dust load that favors safety and health during logistics and use.

Keywords Biochar · Compost · Digestate · Co-composting · Soil amendment

1 Introduction

Sustainable production of biomethane is a key option to substitute conventional natural gas and decarbonize the energy system [1]: anaerobic digestion (AD) is the leading route to generate biogas, which can then be further upgraded to biomethane by CH4 separation. Today, the AD process is a well-mature process, bringing environmental and social benefits at both local and global level [2, 3]: the main co-product of biomass anaerobic digestion is a sludge (digestate), which

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David Chiaramonti david.chiaramonti@unifi.it can be applied to soil for agronomic purposes as an organic amendment. Composting is another well-known pathway to stabilize organic matter of various origins through a biooxidative process [4], which brings benefits as volume reduction, sanitization from pathogens, reduction of liquid contaminants, and economic and environmental returns [5, 6]. In anaerobic digestion plants, the composting stage of the solid fraction of digestate generally occupies large volumes and requires long residence time, in addition to complex logistical steps [7, 8]. The addition of a bulking agent in the compost pile is normally recommended, in particular when substrates as digestates are used. The small particle size of the material generates risks of anaerobic conditions within the pile, leading to the production of undesired phenomena as ammonia volatilization [9, 10].

Biochar is the solid product from lignocellulosic biomass pyrolysis, characterized by a high content of stable C, mostly produced through slow pyrolysis. Biochars from intermediate/ fast pyrolysis and gasification are often discussed in literature, even if these show different characteristics. Biochar is a highly porous material with a wide range of possible uses, including

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sustainable agriculture, as it improves the water-holding capacity and the organic matter content in soil [11–13]. This feature is particularly interesting in marginal lands and regions where rain is scarce, and irrigation is difficult for a number of environmental or economic reasons [13, 14]. Co-composting of organic matter and biochar, if compared to conventional composting, can positively affect the composting residence time, reducing both N-compound losses in the atmosphere and leaching, favoring the microbiological activity and in turn increasing the humification process, eliminating or reducing the need for additional bulking agent [15–18]. Biochar, alone or co-composted, also contributes to long-term atmospheric C sequestration in soil, offering a rather low-complexity solution if compared to most of the available C sequestering state-ofthe-art technologies [19, 20]. Sanchez-Monedero et al. [21] reviewed biochar applications to composting, suggesting application rates at the beginning of the co-composting process: the proposed rate was approximately equal to 10% by weight on dry basis of the composting pile. This amount seemed to optimize the process performances, but the suggested range bringing positive results to the process was indicated among a minimum of 3% to a maximum of 20% w/w on dry basis [21].

This study examines the integration of slow pyrolysis and biomass anaerobic digestion through a pyrolysis and cocomposting experimental investigation. Co-composting [22–26] was performed using solid fraction of digestate from biomass AD, straw, and various rates of biochar. Feedstocks and products were characterized, deriving conclusions for future works and applications.

2 Materials, methods, and analytical results

2.1 Analytical methods and main results of feedstock characterization

Analytical characterization of feedstock and products have been carried out according to the main European methods for biochar and compost as agricultural soil amendments; in particular, the European Biochar Certification (EBC) methods and the European Compost Network Quality Standards (ECN-QAS) [25, 26] were adopted. Feedstock characterization results and standards for analysis used are collected in the supplementary material document (online resource).

COMBI was produced through co-composting blends of biochar with digestate solid fraction, and the addition of a small and variable (for the different cases) amount of cereal straw as bulking agent. Biochar was produced in the oxidative CarbOn pilot plant developed by RE-CORD. CarbOn is a continuous biomass carbonization system based on open top, downdraft technology, operating under oxidative pyrolysis regime. The plant is rated for 50 kg h⁻¹ of biomass with up to 20% *w/w* moisture content at inlet. The reactor is externally insulated and consists of a cylindrical volume where biomass is converted in a controlled oxidative environment in the temperature range of 500–750 °C, with a solid residence time of approx. 3 h in the reactor and 2 h in the cooled discharge. A more detailed description of the process and the pilot plant can be found elsewhere [27, 28]. The feedstock used for the production of biochar for this work was chestnut (*Castanea sativa* Mill.): the main chemical and physical characterization can be found in Table 9 (online resource). The experimental conditions during biochar production in the CarbOn unit are reported in Table 1.

The obtained product was sampled according to EBC standards, and results of the analysis for biochar characterization can be found in Table 10 (online resource).

The characterization of the biochar produced and used in the present study confirms that it qualifies for the EBC premium grade quality. In addition to the evaluation of the total specific surface area (nitrogen-based BET method) for surface and porosity characterization, a density functional theory (DFT) analysis was also performed to assess the pore size distribution. This investigation showed a biochar porosity structure mainly composed by micropores having diameters lower than 2 nm, and mesopores with diameters ranging from 2 to about 4 nm. Detailed results of the DFT analysis can be found in Fig. 3 (online resource).

The biochar is mostly very stable carbon to thermochemical and biological degradation, only marginally subject to mineralization by microorganisms [19, 29]. As reported by Leng et al., biochar fixed carbon (BFC) is closely related to stable C content. Calvelo Pereira et al. showed how the thermogravimetric analysis (TGA) can be a suitable and practical mean to evaluate both the stable and the labile carbon fractions (respectively fixed carbon and volatile matter content of biochar) [19, 30, 31].

Concerning the collection of organic matter (OM) for subsequent co-composting with biochar, digestate was supplied by an industrial anaerobic digestion plant located in the North of Italy, mainly fed with manure as main feedstock. The characterization of the digestate can be found in Table 11 (online resource).

The analysis of the Potential Dynamic Respirometric Index (PDRI) of the digestate suggests a well-stabilized organic matrix available at the outlet of the anaerobic process, collected after mechanical dewatering.

 Table 1
 Experimental conditions for biochar production in the CarbOn pilot unit [27]

Operating condition	Slow oxidative pyrolysis
Inlet feed	50 kg _{w.b.} h^{-1}
Maximum process temperature	550 °C
Residence time	3 h

The water content of the solid fraction of the digestate was also a key parameter to be analyzed: it was measured at 63% *w/w*. According to applicable standards, the presence of pathogens (*Salmonella* spp. and *Escherichia coli*) also need to be assessed, but no biological contamination was detected.

The characterization of the bulking agent cereal straw, which is normally used as horse bedding stable, can be found in Table 12 (online resource).

2.2 Co-composting method and main characterization of windrows/piles

The co-composting process adopted in the present work followed the ECN-QAS recommended procedures [26] and was performed during the summer season in a farm located in Scandicci (Florence), Italy. The experiment duration was 60 days, with no additional curing time also keeping into account the time constraints for the planned soil application operations (November 2018) and field agronomic trials.

The composting system adopted for the present work was a static one, with windrows formed within a farm-greenhouse (Fig. 1), and manually turned twice per week. All windrows were prepared for the test at the same time and in the same environment by the same operators; samples for analysis were taken at day 0 and day 60. Windrows dimensions were approximately 2 m (length) \times 1.6 m (width) \times 0.8 m (height), creating a pile of about 1.5 m³ of volume with a vertical section as similar as possible to a semicircular shape. Windrows were prepared starting from a first layer of digestate and finishing them with digestate covering the entire pile. Biochar and straw layers were separated by digestate layers. At the end of the windrows preparation, all piles accounted for the same volume. This layer configuration lasted until the first turning, which occurred after a week. Temperatures were collected before windrow turning by positioning three probes in each pile at one-third of the vertical section from the soil and at one fourth, half, and three-fourths of the horizontal section. Ambient temperature and humidity were not recorded inside the greenhouse due to practical constraints. However, in Fig. 5 (online resource), the average daily temperature and relative humidity of Scandicci (Florence, Italy) are reported, taking data from the meteorological station managed by SIR (Servizio Idrologico della Regione Toscana), an organizational unit of the Tuscany Region. Daily average temperature and daily average relative humidity values are compared to CD and CB2 average windrow temperature. The daily average values of temperature and relative humidity at the meteorological station can be a good approximation of those at the greenhouse, because of their physical proximity (Scandicci, Florence, Italy), and the greenhouse itself can be considered as an open tunnel (as shown in Fig. 1).

Four different blends were considered for composting, one for each windrow. The rates of biochar were increased from 0 to 15.2% by weight on dry basis, for the reasons explained below related to field rate application. Correspondingly, the rates of straw and digestate were decreasing, keeping the initial goals of four windrows with the same volume. The windrows were named CD (digestate and straw only) CB1 (digestate, straw and 12 kg w.b. of biochar), CB2 (digestate, straw and 18 kg w.b. of biochar), and CB3 (digestate, straw and 24 kg w.b. of biochar), as detailed in Table 2. The 18 kg w.b. rate of biochar was selected for CB2, as it corresponds to an application rate of 3 t ha^{-1} w.b. of biochar for the case of agronomic field trials in 60-m² plots. The 12-kg w.b. and 24kg w.b. rate of biochar for CB1 and CB3, respectively, were used to investigate the effects of different doses of biochar in windrows. The biochar percentage by weight - on dry basisof the starting composting piles-is thus mainly related to the digestate humidity as received.

The measured C/N index of the CD pile was equal to 36.3, close to the optimal value for composting reported in literature [32, 33]; in general, higher C/N values of the initial matrix can lead to extend the duration of the composting process [34]. Compared to the control C/N ratio (i.e., the C/N value for the CD pile), the other piles (CB1, CB2, and CB3) showed higher ratios, increasing with the addition of the biochar, which however gives the main contribution in terms of stable and recalcitrant C. The measured initial moisture content fall within the range indicated by literature, i.e., between 50 and 60% in mass fraction [33, 35, 36]. While the pH of the digestate, the main substrate matrix, was equal to 7.00, the pH of biochar was 7.97; this led to reaching an optimum environment for the

Fig. 1 Farm-scale co-composting site—windrows and piles



Table 2	Initial w	indrows compos	sitions			
		U.M.	CD	CB1	CB2	CB3
Windrow		kg d.b.	160.6	156.5	153.0	149.6
Starting r	noisture	% w/w w.b.	61.6	60.0	59.2	58.3
Biochar c	content	kg w.b.	0.0	12.0	18.0	24.0
Biochar r	ate	% w/w d.b.	0.0	7.3	11.2	15.2
C/N inde	x		36.3	40.4	42.7	45.2

microbiological activity, as also reported by de Bertoldi et al., which explained how pH > 7.5 can lead to higher amount of ammonia volatilization [16, 37].

The presence of coliforms, as *E. coli*, was also investigated in the digestate and the different composted products: this analysis was carried out according to APAT CNR IRSA 7030 F Man 29 2003 method. Finally, humic substances content were assessed according to the Regione Piemonte method C 6.3-1998.

Replications of windrows were not possible due to time, space constraints in the greenhouse, and availability of feedstock on short notice. Nevertheless, COMBI was properly sampled at the end of the co-composting process (it was turned manually by operators twice per week) taking sample material from different sections and all along the length of every windrow: three vertical sections were chosen to collect material for analysis (the ones where temperatures probes are located, as described before). Furthermore, the material collected for every section was taken at different height, considering also the external layer. After the sample homogenization, they were analyzed in our laboratory following relative standards for all the analysis chosen (where triplicates are required in most of the cases).

3 Results and discussion

The average temperature for each windrow, as recorded twice a week, before each turning are reported at Fig. 2. Temperatures trends show that maximum temperatures are all falling in a comparable range and above the 55 °C target. The highest peak temperature was reached by CD (58 °C), while the lowest temperature level was observed for CB3 (55.6 °C). However, CB2 and CB3 reached peak temperatures at least 4 days earlier then control CD and CB1. The biooxidative phase of CB2 and CB3 lasted for ~15 days, while CD and CB1 needed ~19–20 days to reach the same temperature levels.

The range of 52–60 °C is considered as the most appropriate for adequate treatment of the OM [33, 38]. However, ECN-QAS for compost operation quality manual recommends 10 days over 55 °C or 3 days above 65 °C for full



Fig. 2 Average temperatures for each windrow during the co-composting process. The graph shows the peak temperatures reached during the oxidative phase and how the values stabilize gradually to the ambient temperature

sanitization of the substrate from human pathogens in open systems [26].

Nevertheless, even if the peak temperature of 55 °C was reached in each windrow, the duration of this phase lasted for at least 10 days only in the case of CD and CB2. In order to verify the full sanitization of the products, they were all analyzed for biological contamination by *Salmonella* spp. The outcome was positive, with all samples resulting pathogen free. However, due to the nature of the main substrate utilized, digestate from manure, this study also investigated the presence of coliforms (*E. coli*): all products showed coliform concentrations below 10 UFC g⁻¹, excluding the case of CB3, which presented a proliferation of 10⁴ UFC g⁻¹, probably due to a contamination during the co-composting process.

The characterization of the four products according to ECN-QAS quality standards is presented in Table 3.

Two further parameters (impurities, weed seed) were not analyzed (even if recommended by ECN-QAS), as the initial materials were substantially free from these thanks to the intrinsic nature of the feedstocks and the upstream processes (slow pyrolysis for biochar production, and anaerobic digestion for digestate). Furthermore, the experiment was conducted under greenhouse conditions, minimizing the risk of external seed contamination. Plant response, instead, is the subject of ongoing agronomic studies, whose results are not yet published. Maximum particle size was substituted by granulometry distribution analysis, presented in Table 4.

It should be noted that limits concerning OM content, contamination, and inorganic pollutants were met by all blends, which would thus qualify for commercial uses in the EU. Further analysis was however performed to better characterize the products in relation to the initial biochar content of the windrows, in particular as regards the OM transformation (Table 4). The water content of the COMBI blends was reduced by 9.8%, 11.5%, 9.5%, and 5.7% for CD, CB1, CB2, and CB3, respectively, if compared to the initial moisture.

Table 3	COMBI characterization	and including threshold	following ECN-QAS	quality standards [26]
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Parameter	U.M.	CD	CB1	CB2	CB3	Method
Organic matter Limit: OM≥15	% w/w d.b.	75.47	81.34	81.49	82.03	CNR IRSA 2 Q64 Vol 2 2008
Liming value (CaO)	% w/w d.b.	2.67	2.23	1.74	3.36	EPA 3051A 2007 + EPA 6010D 2014
Total N	% w/w d.b.	4.19	2.84	2.69	2.41	EN 15104
Total P	$mg kg^{-1} d.b.$	11,353	7599	2826	2873	EN 15290
Total K	$mg kg^{-1} d.b.$	25,568	22,215	17,405	16,656	EN 15290
Total Mg	$mg kg^{-1} d.b.$	7737	6917	4813	5452	EN 15290
Bulk density	$g l^{-1}$ w.b.	160	160	150	160	EN 15103
Dry matter	% <i>w/w</i> w.b.	48.2	51.5	50.3	48.0	EN 14346
Electrical conductivity	$\mathrm{mS~cm}^{-1}$	3.57	2.47	2.85	2.41	ANPA 9 Man 32,001
pH value		8.2	8.6	8.2	8.3	CNR IRSA 1 Q64 Vol 3 1985
Aerobic biological activity	mg $O_2 k g_{OM}^{-1} h^{-1}$	270	350	< 200	< 200	UNI 11184:2016
Salmonella (absence in 25 g d.b.)		absent	absent	absent	absent	APAT 3 Man 20 2003
Inorganic pollutants	$\mathrm{mg} \mathrm{kg}^{-1} \mathrm{d.b.}$					
Pb < 130		b.d.l.	b.d.l.	b.d.l.	b.d.l.	EN 15290
Cd<1.3		b.d.l.	b.d.l.	b.d.l.	b.d.l.	EN 15290
Cr < 60 Cu < 300		20	7	5	6	EN 15290
Ni < 40		37	24	21	24	EN 15290
Hg<0.45		5	1.2	b.d.l.	b.d.l.	EN 15290
Zn < 600		< 0.1	< 0.1	< 0.1	< 0.1	EPA 3051A 2007 + EPA 1631E 2002
		196	153	143	142	EN 15290

However, no water was added during the experiment and the ambient average relative humidity value of the location area, where the test was carried out, at the end of the experiment was 66.1% (see Fig. 5, online resource).

The stabilization of organic substrates through the composting process is the result of both degradation and humification of the organic matter (OM), leading to a final content of OM in composted material lower than in the initial windrow, which can be considered a measure of the intensity of the composting process [33, 39]. The OM content was

analyzed in all samples, and a mass balance analysis was performed, considering the dry-weight reduction of the windrows, as recommended by M.P. Bernal et al. [33, 40]. Results are presented in Table 5.

As fixed carbon increases with the biochar content in the initial blend, due to the stable and recalcitrant form of C added with biochar, this fixed carbon amount remains substantially the same during the short-time co-composting process (60 days) [19, 29, 31]. Therefore, in order to adopt a measure representative for the OM yield, the amount of biochar-fixed carbon

Table 4	Combi blends-other	
analysis		

U.M.	CD	CB1	CB2	CB3	Method
% w/w w.b.	51.8	48.5	49.7	52.0	EN 14346
% w/w d.b.	55.5	47.8	20.7	19.4	EN 15148
% w/w d.b.	24.2	30.1	29.9	33.8	EN 1860-2
% w/w d.b.	20.3	22.1	20.7	19.4	EN 14775
% w/w d.b.	40.0	45.4	45.2	50.8	EN 15104
% w/w d.b.	0.4	0.3	0.3	0.3	EN 13654-2
% w/w d.b.	39.6	45.1	44.9	50.5	_
>5 mm %wt >2 mm %wt	21.4 13.4	27.5 17.2	21.3 18.5	33.0 15.9	UNI EN 15149
>0.5 mm %wt	42.2	35.1	38.3	32.3	
<0.5 mm %wt	23.0	20.2	21.9	18.9	
	U.M. % w/w w.b. % w/w d.b. % w/w d.b. % w/w d.b. % w/w d.b. % w/w d.b. % w/w d.b. > 5 mm %wt > 0.5 mm %wt < 0.5 mm %wt	U.M. CD % w/w w.b. 51.8 % w/w d.b. 55.5 % w/w d.b. 24.2 % w/w d.b. 20.3 % w/w d.b. 40.0 % w/w d.b. 9.6 > 5 mm %wt 21.4 > 2 mm %wt 13.4 > 0.5 mm %wt 42.2 < 0.5 mm %wt	U.M. CD CB1 % w/w w.b. 51.8 48.5 % w/w d.b. 55.5 47.8 % w/w d.b. 24.2 30.1 % w/w d.b. 20.3 22.1 % w/w d.b. 40.0 45.4 % w/w d.b. 0.4 0.3 % w/w d.b. 39.6 45.1 > 5 mm %wt 21.4 27.5 > 2 mm %wt 13.4 17.2 > 0.5 mm %wt 42.2 35.1 < 0.5 mm %wt	U.M. CD CB1 CB2 % w/w w.b. 51.8 48.5 49.7 % w/w d.b. 55.5 47.8 20.7 % w/w d.b. 24.2 30.1 29.9 % w/w d.b. 20.3 22.1 20.7 % w/w d.b. 40.0 45.4 45.2 % w/w d.b. 0.4 0.3 0.3 % w/w d.b. 39.6 45.1 44.9 > 5 mm %wt 21.4 27.5 21.3 > 2 mm %wt 13.4 17.2 18.5 > 0.5 mm %wt 42.2 35.1 38.3 < 0.5 mm %wt	U.M.CDCB1CB2CB3 $\% w/w$ w.b. 51.8 48.5 49.7 52.0 $\% w/w$ d.b. 55.5 47.8 20.7 19.4 $\% w/w$ d.b. 24.2 30.1 29.9 33.8 $\% w/w$ d.b. 20.3 22.1 20.7 19.4 $\% w/w$ d.b. 40.0 45.4 45.2 50.8 $\% w/w$ d.b. 0.4 0.3 0.3 0.3 $\% w/w$ d.b. 39.6 45.1 44.9 50.5 $> 5 \text{ mm %wt}$ 21.4 27.5 21.3 33.0 $> 2 \text{ mm %wt}$ 13.4 17.2 18.5 15.9 $> 0.5 \text{ mm %wt}$ 42.2 35.1 38.3 32.3 $< 0.5 \text{ mm %wt}$ 23.0 20.2 21.9 18.9

 Table 5
 Product quality comparison: net organic matter (NOM) yield for every blend

		CD	CB1	CB2	CB3
BFC	kg d.b.	0.0	9.2	13.8	18.4
NOM in	kg d.b.	138.9	127.1	120.0	112.9
NOM out	kg d.b.	53.4	53.2	56.5	63.6
NOM yield	%	38.5	41.8	47.1	56.3

(BFC) was subtracted from the total OM content (Table 3), defining the new parameter net organic matter (NOM). NOM at the end of the process, expressed as a percentage of the initial NOM, increased almost linearly with the initial biochar rate added to the compost pile (CB3 > CB2 > CB1 > CD).

A higher percentage of OM in the composted material with higher percentages of biochar could lead to assume that cocomposting negatively influences the intensity of the biooxidative phase, thus lowering the rate of degradation and stabilization of the OM in the initial material. However, three parameters listed below (PDRI, Humification NH₄-N/NO₃-N ratio) show a stabilized compost for all the blends.

The measure of the Potential Dynamic Respiration Index (PDRI) showed better stabilization for CB2 and CB3. Specific oxygen uptake rate is in fact a direct parameter to assess the compost stabilization at the end of the process. Following Bernal et al., which refer to the Californian Compost Quality Council (CCQC) maturity index [33, 41], PDRI values above 1000 mg $O_2 kg_{OM}^{-1} h^{-1}$ are representative of unstable compost, while below 300 mg $O_2 kg_{OM}^{-1} h^{-1}$, the compost is considered as very stable. CB1 showed the highest value, with 350 mg $O_2 kg_{OM}^{-1} h^{-1}$, while the others resulted below the reference limit. However, it should be noted that only CB2 and CB3 showed PDRI 200 mg $O_2 kg_{OM}^{-1} h^{-1}$.

Other parameters can also be investigated in order to evaluate the bio-stabilization level of the products, such as humic acid (HA) and fulvic acid (FA) content, which are representative of the humification degree. A higher degree of humic substances correspond to a more efficient stabilization of the OM during composting [42]. HA and FA are heterogeneous complexes which can be classified by molecular weight, functional groups, and degree of polymerization and cyclization [33, 43, 44]. The four blends were analyzed for humic substance content and the results are shown in Table 6. CB1 content of HA and FA was the lowest observed, although comparable with CD. CB2 values for HA and FA were the highest. CB3 showed the lowest HA value and intermediate FA value. These results suggest that the amount of biochar in the initial windrow does not allow a linear prediction of the HA and FA content, and that the CB2 windrow apparently maximized the synergistic effects of co-composting on the microbial humification processes.

As reported in the table above, the two main indexes used in this study to evaluate the humification level of the four blends, following Roletto et al. [43], were the Humification Index (HI, representing the ratio between HA and organic carbon contents) and the Polymerization Index (PI, representing the ratio between HA and FA). HI, in this paper, was calculated considering NOM as the organic carbon content, thus excluding the fixed carbon content of the amount of biochar used in blends. All products showed HI index higher than the minimum reference threshold, but while the control CD and CB1 showed comparable HI values, CB2 and CB3 showed a humification index at least three times higher than the previous ones. PI resulted below the limit only for the case of CB3, whereas the other samples showed comparable values.

A high level of NH₄-N forms is an indication of a low stabilization for the OM. The fate of nitrogen forms varies along the composting process: the NH₄-N form is prevailing during the mineralization processes of the OM, typical of the bio-oxidation phase.

As shown in Table 7, the NH₄-N contents for all the blends fall below the limit of 0.04% w/w d.b. proposed by Zucconi and de Bertoldi [45] for mature compost (though from the organic fraction of municipal solid wastes), even if the same parameter for CD was at least three times the other blends. On the other hand, since nitrification of ammonium mostly occurs after the thermophilic phase, NO₃-N concentration can also be retained as a good indicator of compost stabilization. Bernal et al. [46] proposed a limit of 0.16 to the NH₄-N/NO₃-N index to define a compost sufficiently mature. All blends remained below the 0.16 limit for the NH₄-N/NO₃-N ratio, with CB2 and CB3 giving the lowest values.

A summary of COMBI characteristics compared to the composted digestate is given in Table 8; the main parameters chosen to evaluate the quality of the materials produced by composting and co-composting were the final biochar rate, the NOM yield, the humified organic matter, the compliance with

Table 6Product qualitycomparison: humification andpolymerization indexes

Parameter	U.M.	CD	CB1	CB2	CB3	Threshold [43]
Humic acid	% w/w w.b.	3.72	3.60	23.66	1.97	_
Fulvic acid	% w/w w.b.	1.54	1.37	7.87	5.39	_
Humification Index (HI)	%	4.0	3.6	20.0	13.2	≥3.5
Polymerization index (PI)	%	2.4	2.6	3.0	0.4	≥ 1

 Table 7
 Product quality direct

 comparison: nitrogen compounds
 (NH₄-N/NO₃-N ratio) in COMBI

 blends and digestate
 (NH₄-N/NO₃-N ratio)

Parameter	U.M.	CD	CB1	CB2	CB3	Method
Total N	% w/w d.b.	4.2	2.8	2.7	2.4	ANPA 13 Man 3 2001
Organic-N/total N	%	99.15	99.65	99.55	99.63	ANPA 14 Man 3 2001
Nitrate-N	% w/w d.b.	0.280	0.100	0.180	0.130	EPA 300.11997
Ammonium-N	% w/w d.b.	0.036	0.010	0.012	0.010	ANPA 14 Man 3 2001
NH ₄ -N/NO ₃ -N ratio		0.129	0.100	0.067	0.077	_
C/N		9.55	15.99	16.80	21.08	_

the ECN-QAS limits for inorganic pollutants, and the product stabilization and sanitization indexes.

4 Conclusions

A production test of COMBI for field trial application was carried out in this study. The control (CD) was prepared by composting solid digestate only with straw; CB2 blend was prepared considering a field application of 3 t ha⁻¹, while CB1 and CB3 blends were prepared with a $\pm 30\%$ *w/w* w.b. respect to the initial biochar content in CB2.

The experimental work led to a biochar concentration in the final product of 14.9, 19.8, 22.8% *w*/w d.b. The difficulty in estimating the final biochar concentration by weight on dry basis during COMBI production was an outcome of the production run. Indeed, when COMBI is directly produced in field conditions, the uncertainties in measuring the moisture content of different samples of the solid fraction of digestate influence the mass yield prediction, which is also dependent on local climatic conditions.

The composting process was carried out with a biochar concentration in the initial blend of at least 11.2% w/w d.b.; CB2 and CB3 ended the bio-oxidative phase about 4 days earlier than CD and CB1. At the same time, biochar addition did not increase the peak temperature of the co-composting

process, as also reported in literature. The process parameter condition (10 days over 55 °C) to guarantee the sanitization, indeed, was obtained only for CD and CB2. However, as regards a potential contamination by pathogens, only CB3 resulted with a *E. coli* proliferation (10^4 UFC g⁻¹).

The compost obtained from the control (CD) met all main reference limits, but products characteristics, in terms of a quantitative comparison with CB2, were always lower, in particular, regarding the product stabilization obtained.

CB2 blend, having an initial biochar concentration of 11.2% w/w d.b., attained a final concentration of 19.8% w/w d.b.: it outperformed the other blends on all process and product parameters, showing the lowest stabilization time, the highest NOM yield with the highest degree of humification, and the lowest ammonium/nitrate ratio index. This result suggests that the 11.2% w/w d.b. initial rate of biochar in the blend maximized the synergistic effect of co-composting the solid fraction of digestate with biochar. In field conditions, especially at larger scale of composting operations (e.g., anaerobic digestion plants), this rate should be further screened to increase process efficiency.

Furthermore, it can be speculated that, if applied to soil as an amendment, CB2 could outperform the other blends in terms of OM increase in soil, considering its humification rate; however, this has to be further investigated in agronomic field trials. Stability of the recalcitrant carbon contained in biochar

 Table 8
 Product quality

 comparison: all the results
 obtained summarized

Parameter	CD	CB1	CB2	CB3
Final biochar rate % w/w d.b.	0	14.9	19.8	22.8
Reduction of processing time	Control	No reduction	$\sim 4 \text{ days}$	$\sim 4 \text{ days}$
Sanitization (10 days over 55 °C)	Yes	No	Yes	No
Pathogens contamination	No	No	No	E. coli
ECN-QAS limit observance	Yes	Yes	Yes	Yes
Net organic matter yield %	38.5	41.8	47.1	56.3
PDRI mg $O_2 kg_{OM}^{-1} h^{-1} (< 300)$	< 300	350	< 200	< 200
Humification Index (HI) (> 3.5%)	4.0	3.6	20.0	13.2
Polymerization index (PI) (> 1.0%)	2.4	2.6	3.0	0.4
NH ₄ -N % <i>w/w</i> d.b. (<0.04%)	0.036	0.010	0.012	0.010
NH ₄ -N/NO ₃ -N ratio (< 0.16)	0.129	0.100	0.067	0.077

can also contribute to the carbon sink of soil for the mitigation of greenhouse gas emissions.

A qualitative result of the experiment which should be highlighted is dust reduction in biochar: after mixing the windrows, the typical black dust that normally develops when handling biochar, could not be visually observed. This represents a tremendous advantage in terms of logistics, handling, health, and safety of biochar, when it is transported, stored and applied to fields.

The results from the present work suggests that the role of biochar in co-composting of digestate is relevant and consistent with literature for other organic substrates. In particular, considering carrying out the operations in field conditions of an anaerobic digestion plant, it is important to underline the benefit observed regarding the time reduction needed by the process, also corresponding to a product with better characteristics than without biochar addition.

Two of the main parameters investigated for COMBI characterization were the biochar rate and the NOM content. An application rate based on weight per hectare on wet basis could be misleading due to the possible uncertainties in sample collection and representativity, as regards the moisture content. The adoption of a volume-based approach, instead, allows for a better evaluation of the actual biochar application rate per hectare.

Further research on the co-composting of biochar and digestate could include mass balance of nitrogen and carbon through measuring of gaseous emissions from windrows, to establish also greenhouse gas emissions of the process. The use of lysimeters is thus recommended for the scope. Since C/ N ratio normally used in agriculture is difficult to apply to co-composted blends with biochar for agronomic application, further investigation on water-soluble C/N of the final product is suggested.

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