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"THE ORIGINALS

Evaluation of two composting strategies for making pig slurry solid fraction suitable for pelletizing

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Abstract

In this study, pig solid fraction (SF) was composted aiming at obtaining a composted manure with a moisture content <40% - suitable for pelletizing. Six identical SF windrows of approximately 4 m^3 and 1800 kg were set up outside, on a concrete pad in an open-sided, roofed facility, and composted for a period of 72 days. During the experimental period, three SF windrows were composted unturned (NTW), while the others three SF windrows were turned (TW) six times: at day 7, 16, 28, 35, 50 and 57. Carbon dioxide (CO_2), methane (CH_4), nitrous oxide (N_2O) and ammonia (NH_3) emissions were measured three times a week for the first 3 weeks and twice per week thereafter for the 72 days of composting. In correspondence of each turning operation, gases emissions rates from TW, were evaluated two times: before and immediately after turning. Due to the production of heat generated during the composting process, high losses of water occurred from both NTW and TW. However, at the end of the trial the average moisture content was significantly lower in TW than in NTW. We conclude that composting of pig SF in turned windrows represents a valuable process to concentrate the nutrients in manure and make it suitable for pelletizing, reducing the moisture content from 73.4% to 34.6%. However, in terms of CO_2-eq, total gaseous emissions recorded over 72 days of trial from TW (120.43 kg CO_2-eq. t^{-1}) were approximately 95% higher as compared to those (64.71 kg CO_2-eq. t^{-1}) obtained from NTW.

Key words: Carbon dioxide; methane; nitrous oxide; ammonia; swine solid fraction.

1. Introduction

In Italy, as well as in many European countries, the pig farming has experienced a major shift toward intensification in recent years (ISTAT, 2012). As a result of intensified livestock operations, huge volumes of liquid (slurry) manure are produced (approx. 17 million tons per year; Colonna and Alfano, 2010), often in far greater amounts than those which can be used in the available farmland where the animals are raised. This could cause adverse environmental consequences (Rao et al., 2007) such as nitrate water pollution both in surface and in ground waters, especially in areas classified as Nitrate Vulnerable Zones in accordance with European Regulation (91/676/CEE). Consequently, there is a growing need to adopt slurry treatment technologies to optimize the management of the large amount of manure generated and to reduce potential risks of environmental pollution.

In this context, the conversion of separated solid fraction (SF) from pig slurry to dried pellets represents an increasingly popular option for farmers in the management of manure. According to Pampuro et al. (2013) this process has the potential to increase the bulk density of SF from an initial value of 400-450 kg m^{-3} to a final one of more than 1000 kg m^{-3}. This allows better handling and transportation of SF at further distance (even at hundreds of km as order of magnitude) in order to move N (nitrogen) from Nitrate Vulnerable Zones to others which are not vulnerable. Furthermore, pelletization could be a valuable process to homogenize, standardize and concentrate the nutrients in the SF, improving at the same time the uniformity of its fertilizing and amending actions (Romano et al., 2014).

A key part of this technology is the composting of fresh SF, in order to stabilize manure (e.g., converting readily available N to more stable organic N) and remove odours and pathogens (Nolan et al., 2011; Parkinson et al., 2004). The heat generated by composting also allows the drying needed to make manure suitable for pelletizing. According to Alemi et al. (2010) the optimal moisture content of SF to be used for pelletization varies between 20% (for a disk pelletter) and 35-40% (for an extruder). Composting of SF involves its storage in a windrow that can be turned or not turned. Frequent turning allows aeration of piles, fragmentation of large particles and reduction of composting times (Szanto et al., 2007). However, composting of manure has been identified as a significant source of harmful gases such as carbon dioxide (CO_2), methane (CH_4), nitrous oxide (N_2O) and ammonia (NH_3) (e.g., Fukumoto et al., 2003; Dinuccio et al., 2008). CO_2, CH_4 and N_2O are greenhouse gases (GHG) and are recognized as contributing to the greenhouse effect (IPCC, 2013). NH_3 can cause eutrophication and acidification of soils (Pearson and Stewart, 1993) and also indirectly contributes to N_2O emissions by increasing the N-cycling in natural ecosystem (IPCC, 2013). Furthermore, high NH_3 and N_2O volatilization impacts negatively the manure composting process, by decreasing N concentration and hence compost quality (Bernal et al., 2009). Some studies investigated gaseous emissions during storage of pig solid manure heaps (e.g., Hassouna et al., 2008; Petersen and Sørensen, 2008; Dinuccio et al., 2012). However, to our knowledge, NH_3 and GHG (N_2O, CO_2 and CH_4) losses during composting of pig slurry SF for pellet production have had limited investigation, and the effect of turning the pig slurry SF during composting on gaseous emissions is also little known.

In this study, pig slurry SF was composted in windrows aiming at obtaining a composted SF with a moisture content < 40% - suitable for pelletizing. The objective of the study was to investigate the effects of two different composting strategies (turned and not turned) on i) manure physicochemical characteristics and ii) NH_3 and GHG (CO_2, CH_4 and N_2O) emissions during composting.

2. Materials and Methods

The trial was carried out at Institute for Agricultural and Earth Moving Machines (IMAMOTER) – Italian National Research Council (CNR) - in Turin, Italy (44°57' N, 7°36' E, 245 m above sea level). The SF used for the experiment was obtained by processing about 100 m^3 of fresh raw slurry from a fattening pig farm, by means of a screw press separator (Chior, mod. COM300/600).

Six identical SF windrows were set up outside, on a concrete pad in an open-sided, roofed facility, and composted for a period of 72 days. Each windrow (1800 kg mass, 4.0 m^3 volume) was trapezoidal in shape, about 6 m in base-length and 1 m in height. During the experimental period, three SF windrows were composted unturned (NTW), while the other three SF windrows were turned (TW) six times: at day 7, 16, 28, 35, 50 and 57.

2.1. Temperature monitoring

The temperature of each SF windrow was continuously recorded with three sets of thermocouple sensors (Type K) connected to a multichannel acquisition system (Grant, mod. SQ 1600). Each set was made up of two thermocouples: one (T1) placed at a depth of 0.2 m and the other one (T2) at 0.6 m from the surface of the windrow. Daily air temperature was also monitored and recorded.

2.2. Manure sampling and analysis

At the beginning and at the end of the trial, samples from bottom, surface, sides and centre of each windrow were taken and mixed together to make them representative of the composting windrow (Getahun et al., 2012). During the experimental period, composite samples were also taken at five random locations from each turned windrow in correspondence of the turning operations. All collected samples were analyzed for pH, moisture content, volatile solids (VS), total organic carbon (TOC), total extractable carbon (TEC), total nitrogen (TN), total ammoniacal nitrogen (TAN), nitrate nitrogen (NO_3-N), C/N ratio and Humification Ratio (HR). Hanna HI 9026 portable pH meter fitted with a glass electrode combined with a thermal automatic compensation system was used to measure pH. Moisture content was measured by drying the samples at 105°C for 24 h. Volatile solids content was calculated based on mass loss after heating at 550°C for 4 h. Samples for TOC analysis were prepared by drying the samples at 105°C for 24 h, followed by treatment with sulphuric acid (H_2SO_4) to eliminate any inorganic C, with subsequent analysis on a C analyzer (Carlo Erba Instruments). Total extractable carbon was determined according to the methodology reported by Romero et al. (2007). Total nitrogen and TAN were determined using the Kjeldahl standard method (AOAC, 1990) and nitrate nitrogen was determined by ion chromatography (Dionex-4000i, Dionex, USA). Humification Ratio was calculated according to the following equation (Roletto et al., 1985):

$$HR\ (\%) = (TEC / TOC) \times 100 \qquad [1]$$

2.3. Gaseous emission measurements

CO_2, CH_4, N_2O and NH_3 emissions from NTW were measured three times a week for the first 3 weeks and then twice a week for the following 7 weeks, for a total of 23 times during the 72-day experimental period. For TW CO_2, CH_4, N_2O and NH_3 emissions were measured according to the same schedule as for NTW. However, in correspondence of each turning operation, gases emissions rates from TW were evaluated before turning and immediately after turning. Therefore, for the latter manure composting strategy, a total of 29 measurement sessions per each investigated gas were completed.

NH_3 emissions were measured by wind tunnels and according to the flux measurement procedure described by Dinuccio et al. (2012). Three wind tunnels per windrow were used. Each wind tunnel covered a surface of 0.32 m^2 (0.80 m length x 0.40 m width). During measurement a fan was linked to the tunnel through a flexible pipe to produce an air flow of about 0.6 m s^{-1} over the emitting surface. Each sampling lasted 24 h. Emissions of CO_2, CH_4 and N_2O from the SF windrows were measured according to the same schedule as ammonia emission measurements, using static closed chamber technique. At each sampling time, three polyvinyl chloride chambers (volume 0.021 m^3, surface area 0.138 m^2) were evenly placed over surface of each investigated windrow. Thirty mL of air was drawn with a plastic syringe from the chamber headspace at 0, 10 and 20 min after chamber placement. According to the experimental design as explained above, a total of 468 gas samples were collected, 261 of which from TW, and 207 from NTW. All samples were stored in airtight glass vials and analyzed for CO_2, CH_4 and N_2O within 24 h by gas chromatography (Agilent 7890). The gas chromatograph (GC) was equipped with a thermal conductivity (TCD), a flame ionization (FID) and an electron capture (ECD) detectors for determination of CO_2, CH_4 and N_2O concentrations, respectively. Helium (He) and a gas mixture of argon and methane (Ar–CH_4) were used as the carrier gas for CO_2/CH_4 and N_2O, respectively. The concentrations of each GHG (CO_2, CH_4 and N_2O) were plotted over time, the data were then fitted to a 1st degree equation (C= ax + b, where C is the concentration of gases and x is the time in minutes) or 2nd degree polynomial equation ($C= ax^2 + bx + c$) (Hutchinson and Livingston, 1993)", the slop of which gave the relative change in concentrations per chamber volume and minute. Gas fluxes F in mg m^{-2} min^{-1} were determined according to:

$$F= (dC_{gas}/dt) \times (V/A) \qquad [2]$$

where:

dC_{gas}/dt is the change of CO_2, CH_4 or N_2O concentration in the chamber in mg min^{-1}; V is the chamber volume in m^3 and A is the area covered by the chamber in m^2. Cumulative emissions were approximated by assuming that daily fluxes represented the average flux between each measurement.

Total gaseous losses were expressed in CO_2-eq using conversion factors of 1, 28, 265 and 2.65, respectively for CO_2, CH_4, N_2O and NH_3 (IPCC, 2013).

2.4. Statistical analysis

Data were subjected to statistical analysis using SPSS software version 21 for Windows (SPSS, 2012). One-way analysis of variance (ANOVA) was performed to compare variations in compost parameters and gaseous emissions. Data distribution normality and assumption of equal variance was checked using the Shapiro-Wilk and Levene test, respectively.

3. Results and discussion

3.1. Composting trial

The air temperature and the temperature inside the windrows as a function of time is shown in Figure 1. The average daily air temperature recorded during the experimental period was 23.5°C, with a measured maximum of 27.8°C and minimum of 18.8°C. The temperature profiles (Figure 1) of the SF windrows followed the expected trends (e.g., Luo et al., 2008; Dinuccio et al., 2012) suggesting that the manure was easily compostable.

The average temperature of SF windrows rose to approximately 55°C during the first 6-7 days from their establishment. After this peak, the average temperature gradually decreased to approximately 40.0°C at day 45 and day 55, respectively, for NTW and TW, indicating the end of the thermophilic phase (Bernal et al., 2009). In TW the average temperature rose again after turning operation (Figure 1) due to the increased levels of oxygen that stimulate microbial activity (Getahun et al., 2012). However, from day 55 until the end of the experiment (day 72) the average daily temperatures measured inside TW resulted not significantly different ($p>0.05$) than those recorded in NTW. The highest temperatures were measured at 0.6 m depth in all windrows, with maximum recorded values of 65.9°C and 70.1°C, respectively, for NTW and TW.

The solid fraction of pig manure was characterized by an initial moisture content of 73.4% (Table 1), above the optimal range of 50-60% (Bernal et al., 2009). Similar values were found by Chiumenti et al. (2007) during composting of pig manure. This characteristic could negatively affect air diffusion into the windrow by the chimney effect, a relevant aspect during the thermophilic phase (Chiumenti et al., 2007). As expected, due to the production of heat during the composting process (Figure 1), high losses of water occurred from both NTW and TW (Table 1, Figure 2A). However, at the end of the trial the average moisture content was significantly ($p<0.05$) lower in TW than in NTW (34.6 % vs 46.7 %, $p<0.05$) (Table 1), consistent with the significantly ($p<0.05$) higher average temperature recorded over the trial in TW than in NTW.

The pH decreased during the composting process in both TW and NTW (Table 1), in agreement with the findings from other studies (e.g., Ko et al., 2008; Ogunwande et al., 2008; Getahun et al., 2012). However, TW maintained pH alkaline (range 7.5–8.0) during composting (Figure 2B), while NTW strategy significantly ($p<0.05$) reduced pH from alkaline to slightly acidic (8.4 at day 0 vs 6.9 at day 72) (Table 1). A likely explanation is the production of organic acids in anaerobic conditions caused by the absence of turning operations (Shen et al., 2011).

TOC content also decreased over time (Table 1, Figure 2C) due to the degradation of organic material (Getahun et al., 2012), whereas VS (Table 1, Figure 2D) and TN (Table 1, Figure 2E) concentration increased substantially due to the concentration effect caused by the high water evaporation (Bernal et al., 2009). Similar results were found by Paredes et al. (1996) during composting of different organic wastes. Specifically, relative to the fresh SF (day 0), the concentration of VS and TN in the composted manure at the end of the trial (day 72) resulted, respectively, 1.25 and 3.60 times higher for NTW, and respectively 1.31 and 4.25 times higher for TW (Table 1). After 72 days of composting (end of trial) the concentration of TOC and TAN resulted significantly ($p<0.05$) higher in TW than in NTW (Table 1).

Stability and quality of the final products

As stated by Bernal et al. (2009), the principal requirement of a compost for it to be safely used in soil is a high degree of stability or maturity, which implies a stable Organic matter content and the absence of phytotoxic compounds and plant or animal pathogens. Maturity is associated with plant-growth potential or phytotoxicity (Iannotti et al., 1993), whereas stability is often related to the compost's microbial activity (Bernal et al., 2009). However, both stability and maturity usually go hand in hand, since phytotoxic compounds are produced by the microorganisms in unstable composts (Zucconi et al., 1985). For this reason compost maturity and stability are often used interchangeably. Chemical methods, including C/N ratio in the solid phase (Bernal et al., 1998) and nitrification (TAN and nitrate nitrogen concentration; Ko et al., 2008) are generally used to assess the degree of compost maturation.

In our study, the C/N ratio decreased from 33.3 at day to 12.0 and 10.8 at 72 days for NTW and TW, respectively (Table 1, Figure 2H). Previous research showed that a C/N ratio below 20 was assumed to be indicative of maturity compost (Bernal et al., 1998; Ko et al., 2008).

Ammonium and nitrate nitrogen concentrations provided some indication of compost maturity, because the concentration of nitrate should be higher than that of ammonium at the end of composting process. As shown in Table 1, at the end of the experimental period, the concentration of NO_3-N was higher than that of TAN for TW, while, for NTW, we found a concentration of NO_3-N lower than that of TAN. In TW, the initially high ammonium level dropped rapidly during composting (Figure 2F), while nitrate concentration increased significantly after 36 days (Figure 2G). This result could be explained by the fact that bacteria responsible for nitrification were strongly inhibited by temperature greater than 40°C (Jimenez and Garcia, 1989), so nitrate concentration did not change greatly during the active composting stage.

The evaluation of the humification degree of the OM during composting is an agronomic criterion for compost quality. The agricultural value of a compost increases when the OM reaches a high level of humification. As stated by Senesi (1989), the humification of the OM during composting is revealed by the formation of humic acids. During composting, humic substances (C_{EX}) are produced and humic acid-like organic-C (C_{HA}) increases, while fulvic acid-like organic C (C_{FA}) decrease due to microbial degradation. According to Bernal et al. (2009) an HR higher than 7 indicates a good degree of humification: at the end of the composting process NTW achieved HR equal to 13.7, while TW was characterized by HR equal to 15.1.

3.2. Gaseous emissions

In agreement with other published data (e.g., Sommer and Moller, 2000; Dinuccio et al., 2012), the emissions of CO_2, CH_4, N_2O and NH_3 took place primarily during the thermophilic phase (Figure 3), when microbial activity reached its maximum.

The patterns of CO_2 emission (Figure 3A) observed in this study resemble those reported by Chiumenti et al. (2007). Specifically, CO_2 emission rates were significantly ($p<0.05$) greater for TW than NTW during early composting (day 0–37), but there were no significant ($p>0.05$) difference during late composting (day 42–72). Total CO_2 emissions recorded over 72 days of trial were approximately 2 times higher from TW than NTW (Table 2), reflecting the highest microbial activity and organic matter degradation (Table 1).

The dynamic of CH_4 emissions was also influenced by turning operations (Figure 3B). As expected, CH_4 emission rate decreased immediately after turning operations as a result of the adequate aeration of the windrows (Figure 3B). However, in the interval between turnings, the intense aerobic activity determined a decrease of oxygen concentration that promoted anaerobic conditions and hence the production of methane (Sommer and Moller, 2000; Chiumenti et al., 2007). Fukumoto et al. (2003) showed that the size of the anaerobic portion established inside the compost piles decreased and ultimately disappeared as the composting material matured. In this study, CH_4 emission in TW decreased earlier than in NTW (Figure 3B), suggesting that in TW the length of time until the anaerobic portions disappeared was probably smaller. In spite of this, cumulative CH_4 emissions from the tested manure were not affected significantly ($p>0.05$) by turning operations (Table 2). Total CH_4 emissions represented a loss of 0.29% and 0.30% of the initial VS content in manure (Table 2) for TW and NTW, respectively; values that agreed well with those reported by Hansen et al. (2006) from storage of uncovered pig slurry SF piles.

The production of N_2O (Figure 2C) indicated that during composting, incomplete nitrification/denitrification processes - that normally convert NH_4 into N_2, a non-polluting gas (Yang et al., 2013) - occurred. On average, N_2O flux rates from TW and from NTW were, respectively, 11.1 (range 0.99–60.1) and 1.72 (range 0.29–3.69) mg N_2O m^{-2} h^{-1}. The total nitrogen loss via N_2O pathway from TW accounted for almost 1% of the initial nitrogen content in manure (Table 2). Turning the material significantly ($p<0.05$) increased N_2O emission rates (Figure 2C), probably because NO_2^-/NO_3^- produced in O_2 sufficient region was transferred to O_2 deficient region, thereby promoting N_2O production by denitrification process (Fukumoto et al., 2003). Similar high N_2O emission rates from active aeration composting systems were also observed by Abd El Kader et al. (2007) and by Ahn et al. (2011). As can be seen in Figure 3D, NH_3 emissions across all treatments peaked at the beginning of the trials and then decreased rapidly, resulting in very low values within 7 days. After the first turning operation, NH_3 emissions from TW increased to an average value of 0.11 gNH_3 m^{-2} h^{-1} (Figure 3D). Thereafter, with the exhaustion of easily degradable materials, the degradation rate decreased and consequently the NH_3 emission declined to negligible values (Jiang et al., 2013). While not significant ($p>0.05$), cumulative NH_3 emissions recorded from TW, expressed as g NH_3 t^{-1} of fresh manure, were 11.2% higher than those recorded from NTW (Table 2). The cumulative N losses as NH_3 recorded over the experimental period (Table 2) were in a good agreement with the overall losses (6.5-7.3%) from the initial TN content recorded by Hassouna et al. (2008) during uncovered storage of turned and unturned pig slurry solid manure heaps.

3.3. Mass balance

The data in Table 3 show the quantities of the swine SF at the beginning and at the end of the composting process, considering total solids, volatile solids, total nitrogen and total organic carbon.

The results show a mass loss of 66% and 78% of the initial manure for NTW and TW, respectively. These losses are mainly related to the decrease of moisture (Table 1) that took place during the composting process. In terms of dry matter, 32% (NTW) and 46% (TW) of the initial quantity was lost, while a VS losses of 15.4% (NTW) and 28.7% (TW) were calculated. These results are in line with the higher carbon emissions as CO_2 observed from TW than from NTW.

The carbon losses represented a loss of 64% (NTW) and 85% (TW) of the initial carbon content of the solid manure. Carbon losses from NTW were in a good agreement with the observations of Chiumenti et al. (2007), while the carbon losses from TW were higher than those found by this author.

We calculated the TN balance and found a difference in TN recovery of 11 and 42% of the initial TN content for NTW and TW respectively. The measured nitrogen gas emissions (NH_3 and N_2O) represent 6.3% (NTW) and 8.4% (TW) of the initial nitrogen content. The fraction of nitrogen unaccounted for (4.7% and 33.6% for NTW and TW respectively) can be associated with N_2 emissions (Hassouna et al., 2008). Leached nitrogen and the measurement error may also partly explain this difference. Nitrogen losses of NTW (11% of the initial nitrogen content) and of TW (42% of the initial nitrogen content) were, respectively, in the lower and higher range of the values given by Moller et al. (2000), which varied between 10 and 42%.

4. Conclusions

In accordance with the results of the investigation we conclude that composting of pig SF in turned windrows represents a valuable process to concentrate the nutrients in manure and make it suitable for pelletizing, reducing the moisture content from 73.4% to 34.6%. However, in terms of CO_2-eq, total gaseous emissions recorded over 72 days of trial from TW (120.43 kg CO_2-eq. t^{-1}) were approximately 95% higher as compared to those (64.71 kg CO_2-eq. t^{-1}) obtained from NTW. However a comprehensive assessment requires a whole-system approach which considers not only composting but also emissions after soil application. Experiments under laboratory-scale conditions will be conducted on the application of pelletized composted manure.

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Table captions

Table 1. Chemical characteristics of fresh pig slurry solid fraction (Fresh SF) and of final composts obtained with two turning strategy (NTW=not turned windrows; TW=turned windrows). Mean value and standard error (in parentheses) of 3 replicates.

Table 2. Cumulative emissions of CO_2, CH_4, N_2O, and NH_3 recorded from turned (TW) and not turned (NTW) pig slurry solid fraction windrows. Mean value and standard error (in parentheses) of 3 replicates.

Table 3. Mass balance of the swine manure composting process (NTW=not turned windrows; TW=turned windrows). Mean value of 3 replicates.

Table 1.

Parameter	Fresh SF	Final Compost	
		NTW	TW
Moisture (%)	73.4^{a} (0.7)	46.7^{b} (1.8)	34.6^{c} (0.3)
pH	8.4^{a} (0.2)	6.9^{b} (0.1)	7.6^{ab} (0.2)
VS (%)	27.5^{a} (0.7)	34.3^{b} (0.7)	36.2^{b} (1.2)
TOC (%)	42.3^{a} (0.8)	38.1^{b} (0.4)	36.5^{c} (0.3)
TN (%)	0.8^{a} (0.1)	2.9^{b} (0.1)	3.4^{c} (0.1)
TAN (mg kg^{-1})	2643^{a} (115.7)	959^{b} (20.7)	570^{c} (6.9)
NO_3-N (mg kg^{-1})	1.3^{a} (0.1)	714^{b} (10.5)	1358^{c} (35.5)
C/N	33.3^{a} (1.6)	12.0^{b} (1.2)	10.8^{b} (1.4)
HR (%)	N.D.	13.7 (0.2)	15.1 (0.4)

In the same line, values with different letters are statistically different at $p<0.05$.

Table 2.

Gas	Units	TW		NTW	
CO_2	g t^{-1}	61,196.05^{a}	(4,009.4)	34,711.19^{b}	(1,092.3)
	% VS	22.25	(1.5)	12.62	(0.4)
	kg CO_2-eq. t^{-1} (A)	61.20	(4.0)	34.71	(1.1)
CH_4	g t^{-1}	792.39^{a}	(155.8)	811.52^{a}	(40.3)
	% VS	0.29	(0.06)	0.30	(0.01)
	kg CO_2-eq. t^{-1} (B)	22.19	(4.4)	22.72	(1.1)
N_2O	g t^{-1}	134.42^{a}	(4.3)	22.61^{b}	(5.2)
	% TN	1.68	(0.1)	0.28	(0.1)
	kg CO_2-eq. t^{-1} (C)	35.62	(1.1)	5.99	(1.4)
NH_3	g t^{-1}	537.08^{a}	(22.1)	482.96^{a}	(79.0)
	% TN	6.71	(0.3)	6.04	(0.9)
	kg CO_2-eq. t^{-1} (D)	1.42	(0.1)	1.28	(0.2)
Tot kg CO_2-eq. t^{-1} (A+B+C+D)		120.43^{a}	(7.2)	64.71^{b}	(1.4)

In the same line, values with different letters are statistically different at $p<0.05$.

Table 3.

Parameter	Beginning of the process	End of the process					
		NTW			TW		
		Remaining	Lost		Remaining	Lost	
Initial load (kg)	1800	610	1190	66%	396	1404	78%
Total solids (kg)	478.8	325	153.8	32%	259	219.8	46%
Volatile solids (kg)	131.7	111.5	20.2	15.4%	93.8	37.9	28.7%
TOC (kg)	761.4	274.3	487.1	64%	144.5	646.9	85%
TN (kg)	23.4	20.9	2.5	11%	13.5	9.9	42%

Figure captions

Figure 1. Average environmental temperature and temperatures development at a depth of 0.2 m (T1) and 0.6 m (T2) inside the not turned (NTW) and the turned (TW) windrows (n=3). In TW, arrows mark time of turning.

Figure 2. Changes in content of moisture (A), pH (B), TOC (C), VS (D), TN (E), TAN (F), NO_3-N (G) and C/N (H) of the pig solid fraction during composting in TW. Error bars indicate standard error (n=3).

Figure 3. Emission fluxes of CO_2 (A), CH_4 (B), N_2O (C) and NH_3 (D) during the composting of pig slurry solid fraction in turned (TW) and not turned (NTW) windrows.

Error bars indicate standard error (n=3). In TW, arrows mark time of turning.

Figure 1.

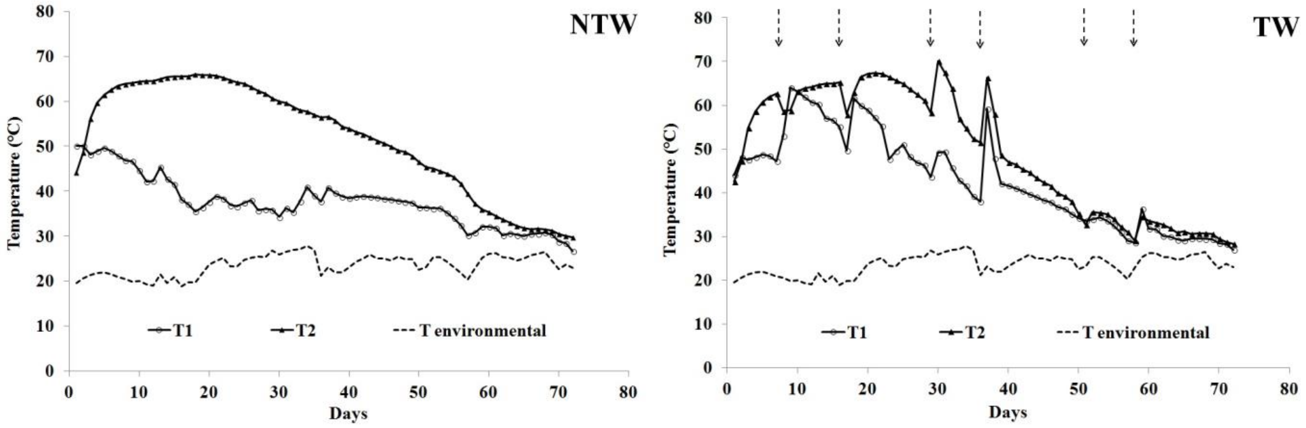

NTW
TW
Temperature (°C)
Days
0
10
20
30
40
50
60
70
80
T1
T2
T environmental

Figure 2.

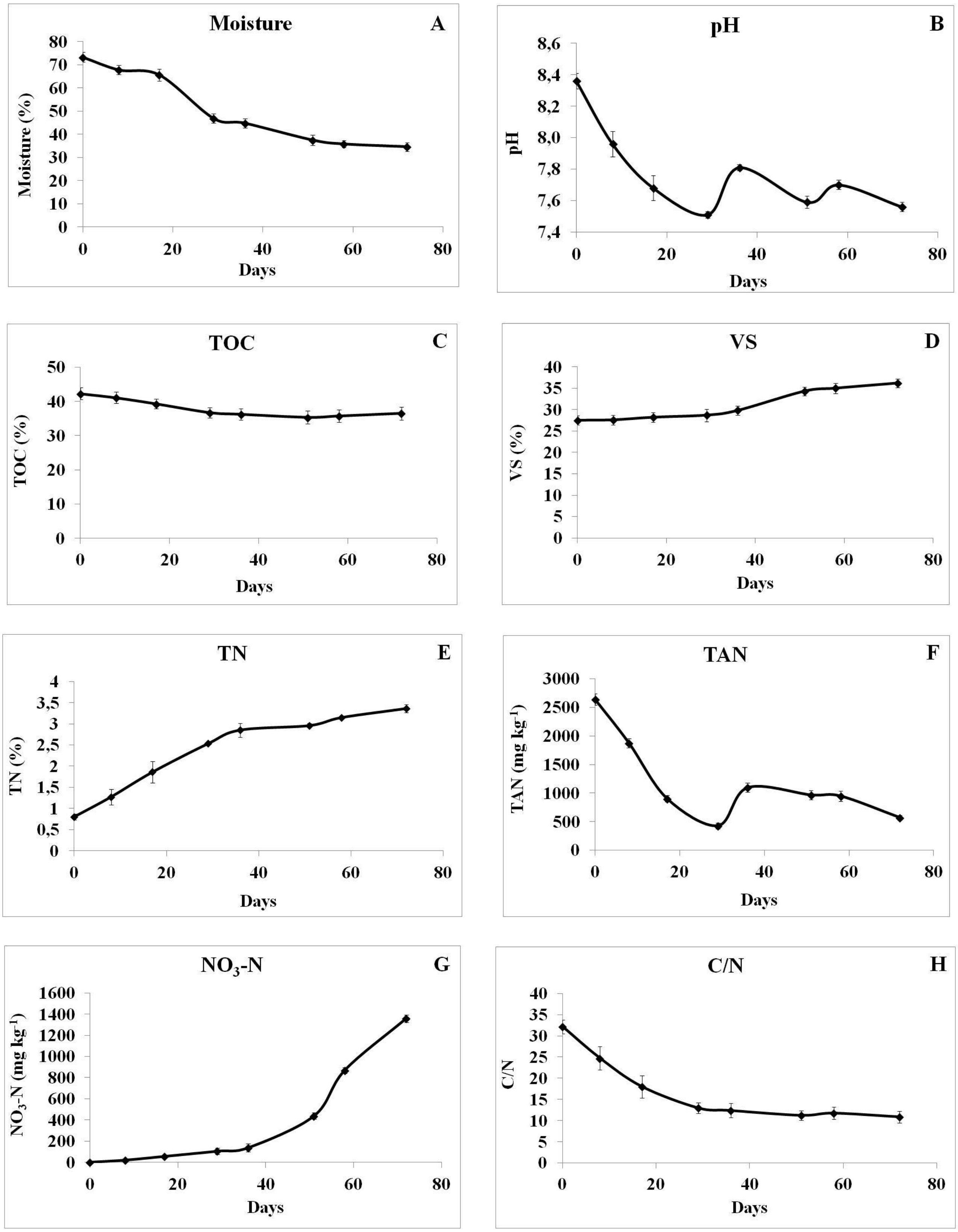
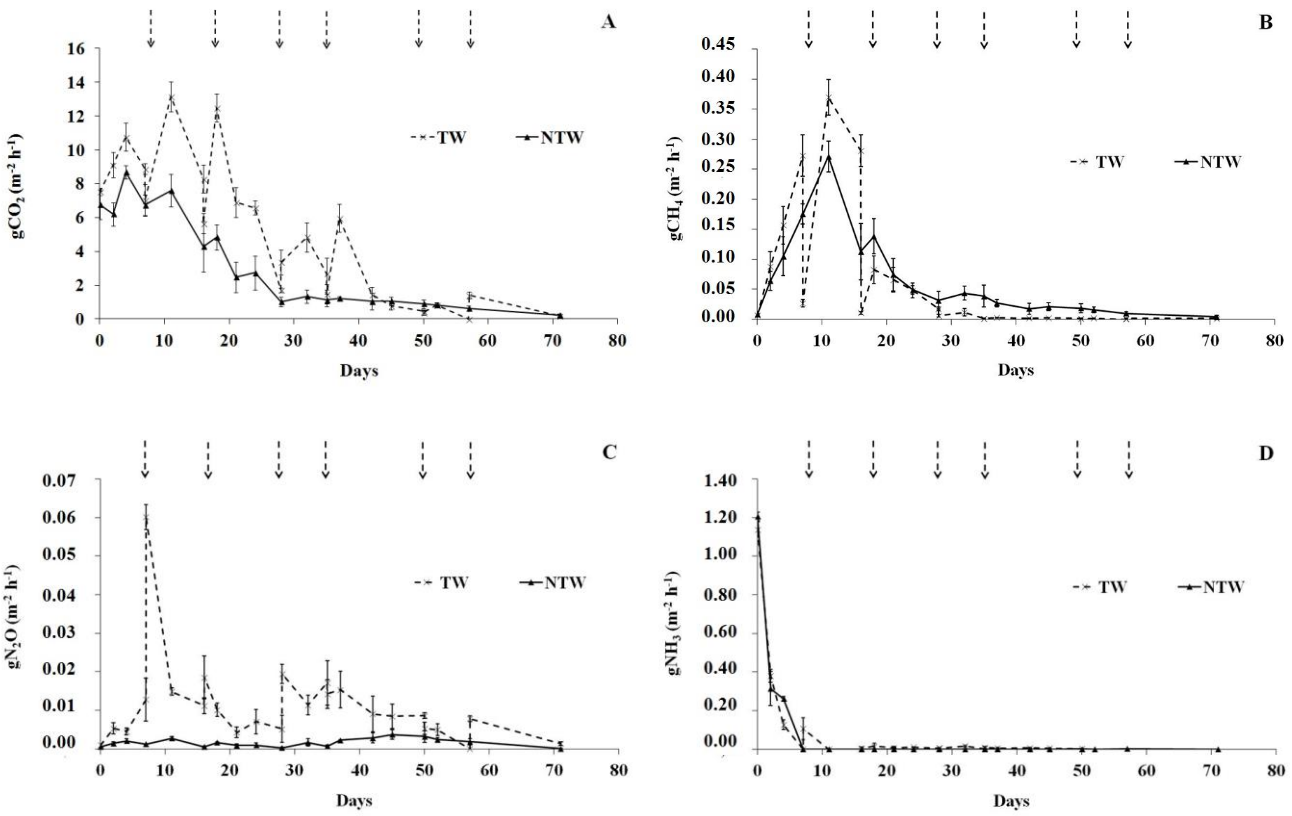

Moisture
A
Moisture (%)
0
10
20
30
40
50
60
70
80
Days
0
20
40
60
80
pH
B
pH
7,4
7,6
7,8
8,0
8,2
8,4
8,6
Days
TOC
C
TOC (%)
0
10
20
30
40
50
Days
VS
D
VS (%)
0
5
10
15
20
25
30
35
40
Days
TN
E
TN (%)
0
0,5
1
1,5
2
2,5
3
3,5
4
Days
TAN
F
TAN (mg kg-1)
0
500
1000
1500
2000
2500
3000
Days
NO3-N
G
NO3-N (mg kg-1)
0
200
400
600
800
1000
1200
1400
1600
Days
C/N
H
C/N
0
5
10
15
20
25
30
35
40
Days

Figure 3.


A
gCO$_2$ (m^{-2} h^{-1})
0
2
4
6
8
10
12
14
16
TW
NTW
Days
0
10
20
30
40
50
60
70
80
B
gCH$_4$ (m^{-2} h^{-1})
0.00
0.05
0.10
0.15
0.20
0.25
0.30
0.35
0.40
0.45
TW
NTW
Days
C
gN$_2$O (m^{-2} h^{-1})
0.00
0.01
0.02
0.03
0.04
0.05
0.06
0.07
TW
NTW
Days
D
gNH$_3$ (m^{-2} h^{-1})
0.00
0.20
0.40
0.60
0.80
1.00
1.20
1.40
TW
NTW
Days

Manuscript ID: APR-D-15-00066
Title: "Composting as a solution to make pig manure suitable for pelletizing: environmental impact of the process."

RESPONSE TO EDITOR AND REFEREES' COMMENTS:
Below are our responses (in **BOLD** type) to the Reviewers' comments

Editorial Notes:

1. List of references should be formatted according to "Guide for Authors".
a) Please provide following reference in English.
a. Colonna and Alfano, 2010.
Response 1: done.

2. Missing reference: The following reference is cited in the text but is missing from the reference list. Please add this missing reference to the list or delete it from the text.
a) AOAC, 1990.
Response 2: done.

Reviewers' comments:

Reviewer #1:

My only reservation was not being used a multivariate statistical technique to analyze the data, such as a correlation matrix and the presentation of data in the form of dendograms classified by Euclidean distances.

> **Response 1: The goal of this study was to investigate the effect of two composting strategies (turned and not turned) on compost physicochemical characteristics and gaseous emissions during composting. Our intention was not to study how the investigated variables are related to one another. For such a reason we consider the used one-way analysis of variance (ANOVA) as the more appropriate statistical method to compare the differences in results**

Use IPCC 2014 data.

> **Response 2: we did not find IPCC 2014 data. However, following your suggestion, we have recomputed the GWPs of CH_4, N_2O and NH_3 with the new values as contained in the 2013 IPCC Guidelines and revised the manuscript accordingly. Specifically we have:**

- **replaced the sentence *"Total gaseous losses were expressed in CO_2-eq using conversion factors of 1, 25, 298 and 2.98, respectively for CO_2, CH_4, N_2O and NH_3 (IPPC, 2007)"* from L43 to L45, page 6, of the original manuscript with the following "Total gaseous losses were expressed in CO_2-eq using conversion factors of 1, 28, 265 and 2.65, respectively for CO_2, CH_4, N_2O and NH_3 (IPCC, 2013)".**
- **revised Table 2**

Reviewer #2

The work reported however is not in line with the introduction and the title as it reports the comparison of two composting strategies of the solid fraction obtained after separation of pig manure.

> **Response 1: Following your suggestion, the title of the paper has been revised to "Evaluation of two composting strategies for making pig slurry solid fraction suitable for pelletizing".**

The results reported not always are easy to understand and in some case the data are not fully reported like the variability of the samples taken in different locations of the windrow.

> **Response 2: we carefully proofread the text to make the "Results and discussion" section more understandable to the reader. We also reported standard error bars in order to show the variability of data depicted in Figure 2. However, we are not able to report the variability of the samples taken in different locations of the windrow. As stated in the paper (page 5, lines 7-14), *"...samples from bottom, surface, sides and centre of each windrow were taken and mixed together to make them representative of the composting windrow (Getahun et al., 2012)".* Therefore at each sampling occasion we analysed one (composite) sample of manure per windrow. To clarify this aspect the sentence *"All collected samples were analyzed for.."* has been replaced with "All composite samples were analyzed for.."**

The methodology used to compare data statistically is not reported and can be just guessed from the results.

> **Response 3: we have now added to the M&M section a new Subsection (2.4. Statistical analysis) to better explain the adopted statistical analyses methodology and clarified how the data were processed. A reference (SPSS, 2012) was also added to the text.**

The 1st or 2nd order fitting of gas concentration is missed in the results.

> **Response 4: we think that this information should be given in the "M&M" section. We have now revised the sentences "The concentrations of each GHG (CO_2, CH_4 and N_2O) were plotted over time, the data were then fitted to a 1st order (linear) or 2nd order (curve-linear) polynomial,.." as follow:**
>
> **"The concentrations of each GHG (CO_2, CH_4 and N_2O) were plotted over time, the data were then fitted to a 1st degree equation ($C= ax + b$, where C is the concentration of gases and x is the time in minutes) or 2nd degree polynomial equation ($C= ax^2 + bx + c$) (Hutchinson and Livingston, 1993)"**

It is quite strange that figure 2 reports only one of the two conditions tested.

> **Response 5: As stated in the manuscript (P.5, lines12-14): "During the experimental period, composite samples were…taken …from each turned windrow in correspondence of the turning operations." For such a reason Fig. 2 depicts only changes in content of the considered physicochemical parameters (moisture, TOC, VS, ………..) in manure during composting in Turned Windrows. It was part of our experimental design to sample and analyse NTW only at the beginning and at the end of the trial (P.5, lines12-14), in order to not affect the composting process. However, we reported the chemical characteristics of fresh manure and those of final composts obtained with the two turning strategy (NTW, TW) in Table 1.**

In any case, an assessment of the environmental impact of the two composting strategies requires, as the authors state at line 12 of page 10, a whole scale approach. The suitability of the compost to be pelletized cannot be state just by the moisture content…

Response 6: we agree. We have now provided more information in the manuscript about the characterization of composted manure. In detail we have:

- **replaced the sentence "All collected samples were analyzed for pH, moisture content, volatile solids (VS), total organic carbon (TOC), total nitrogen (TN) and total ammoniacal nitrogen (TAN)." from L43 to L45, page 6, of the original manuscript with the following "All collected samples were analyzed for pH, moisture content, volatile solids (VS), total organic carbon (TOC), total extractable carbon (TEC), total nitrogen (TN), total ammoniacal nitrogen (TAN), nitrate nitrogen (NO_3-N), C/N ratio and Humification Ratio (HR)".**
- **added to the text in the Abstract section of the revised version of the manuscript: Total extractable carbon was determined according to the methodology reported by Romero et al. (2007). … and nitrate nitrogen was determined by ion chromatography (Dionex-4000i, Dionex, USA). Humification Ratio was calculated according to the following equation (Roletto et al., 1985):**

  $$HR (\%) = (TEC / TOC) \times 100$$

- **added a new Subsection "Stability and quality of the final products" to the Results and Discussion section**

……and the environmental impact need more comprehensive approach. For example the energy used to turn the windrow should be considered.

Response 7: we agree. However energy consumption was beyond the scope of this study. In the M&M section, we now clearly state that:

"The objective of the study was to investigate the effects of two different composting strategies (turned and not turned) on i) manure physicochemical characteristics and ii) NH_3 and GHG (CO_2, CH_4 and N_2O) emissions."

In addition we have replaced the sentence "However a comprehensive assessment requires a whole-system approach which considers not only composting but also emissions after soil application. Experiments under laboratory-scale conditions will be conducted on the application of pelletized composted manure." with the following:

"However a comprehensive assessment of the process requires a whole-system approach which considers not only emissions during composting but also emissions after soil application of pelletized composted manure, as well as the environmental impact of energy consumption. All these aspects are under study by our research group.

Moreover, there is a serious concern on the evaluation of the composting process carried out. The authors do not report any evaluation of the process and the stability of the final product has not been tested. Therefore it is not clear if the microbial activity has been limited by the low humidity or effectively because the active phase was completed. The curing phase has not been considered, anyway.

Response 8: these aspects are now discussed in the Subsection "Stability and quality of the final products"

Finally, I have serious doubt that the composting process can be really carried out with a solid fraction at low dry matter content (26.60%) without the addition of bulking agents.

Response 9: data reported in Table 1 suggest that at the end of the trial the TW reached a good degree of stability and maturity. In particular, the C/N ratio resulted in the range 10-15 (Gomez-Brandon et al., 2008) and the HR value was higher than 7 (Bernal et al., 2009). In addition at the end of the composting process the content of NO_3-N resulted higher than that of TAN, indicating that the process has been

performed under adequate aeration conditions (Bernal et al., 1998). All these aspects are now stated in the manuscript.

Reviewer #3

The objective of the paper is composting for pelletizing, but only the composting process is described. If the objective is pelletizing, the reason for composting has not been clearly stated, as drying may be sufficient for the physical stabilization. The objective should be re-written in order to match the actual content of the paper.

> **Response 1: in the Introduction section (lines 34-41, page 3) of the original manuscript we have specified that: A key part of this technology is the composting of fresh SF, in order to stabilize manure (e.g., converting readily available N to more stable organic N) and remove odours and pathogens. The heat generated by composting also allows the drying needed to make manure suitable for pelletizing. In this work we didn't studied the pelletization process which was investigated in another study (Pampuro et al., 2013; cited in the text).**

In general, the paper should improve the discussion section, finding scientific explanations for the results based on the biological process that occur during composting, taking into account the different composting system used in the two piles.

> **Response 2: we revised the discussion section according to your suggestion**

Information on gaseous emissions during composting of animal manure should be included.

> **Response 3: we have reported information on gaseous emissions during composting of animal manure in the Introduction section (from P.3, line 51 to P.4, line 10).**

The number of gas samples should be clearly indicated. How many samples were taken? How many times per week and how long for?

> **Response 4: to clarify these aspects we have:**
>
> - **replaced the sentence reported in the original paper at page 5, lines 41-45, with: "CO_2, CH_4, N_2O and NH_3 emissions from NTW were measured three times a week for the first 3 weeks and then twice a week for the following 7 weeks, for a total of 23 times during the 72-day experimental period. For TW CO_2, CH_4, N_2O and NH_3 emissions were measured according to the same schedule as for NTW. However, in correspondence of each turning operation, gases emissions rates from TW were evaluated before turning and immediately after turning. Therefore, for the latter manure composting strategy, a total of 29 measurement sessions per each investigated gas were completed."**
> - **added the following sentence to M&M section: According to the experimental design as explained above, a total of 468 gas samples were collected, 261 of which from TW, and 207 from NTW.**

Include here the methodology for calculating total gas emissions for TW and NTW. In TW a very important part of the gases will be emitted during turning operations. Then, the procedure for their estimation must be included.

> **Response 5: we have now explained how cumulative emissions were estimated in M&M section. The following sentence has been added: "Cumulative emissions from each manure windrow over the composting period were estimated, by averaging net flux rates between two sampling points and multiplying by the time interval between sampling points."**

This section needs updated references on manure composting. There is a description of the results during composting, but they require scientific explanation based on references.

Response 6: we agree. Some references on manure composting have been added.

P.7, lines 24-27. The T1 and T2 in Figure 1 cannot be identified. Use different lines and symbols.

Response 7: Figure 1 has been modified according to your suggestion.

The conjunction of high temperature and pile turning was responsible for water evaporation (not only the temperature) as demonstrated by the most effective drying in TW. This needs to be stated in P.7, lines 29-41.

Response 8: we agree. The following sentence has been added to the text: "This difference is probably do to the turning operations which have a positive effect on compost aeration and hence on temperature increase."

Also, the concentration effect of TN can occur during composting due to the mass loss of the pile caused by the organic matter degradation. Then, a mass balance should explain TN losses and conservation in the compost according to the system used. These should be clearly stated in P.7, lines 39-53.

Response 9: we agree. As suggested a mass balance has been conducted to determine the overall effect of composting on manure properties and N-losses. These aspects are now discussed in the Section "3.3. Mass Balance."

In P.7, line 51, the authors said that "the concentration of TOC and TN resulted significantly higher in TW than in NTW" but according to the results shown in Table 1, the values were greater for NTW than for TW.

Response 10: the sentence has been rephrased

P.7, lines 53-56.The limited aeration, which may have led to anaerobic conditions inside the pile, may have limited the biological activity of the aerobic microorganisms responsible for the degradation of the organic matter in NTW. Then, the microbial stability of the compost should be tested or at least some maturation indices presented.

Response 11: we agree. According to your suggestion we have now provided information concerning the content of nitrate nitrogen (NO_3-N), C/N ratio and Humification Ratio (HR) of composted manure. Subsection "Stability and quality of the final products" has been added.

Gas samples were taken before and after each turning in TW, but I cannot see these results. The lines and symbols for TW and NTW in figure 3 must CLEARLY identify each pile (they are too similar).

Response 12: Figure 3 has been modified according to your suggestion

The CO_2 emissions reflect the degradation of the organic matter during composting, so lower values in NTW than in TW are in concordance with previous findings (VS, TOC, etc.). Please, discuss properly.

Response 13: to clarify this aspect, the following sentence has been added to the text (3.3. Mass Balance): "These results are in line with the higher carbon emissions as CO_2 observed from TW than from NTW".

P.8, lines 58-58. The sentence "…CH_4 emission in TW decreased earlier than in NTW…", I cannot see that in figure 3.

Response 14: the sentence has been deleted

Authors should explain how the total gaseous emissions for TW were calculated, as most emissions probably occurred during turning, when measurements cannot be done.

Response 15: please see Response..

Also the authors should take into account the reduction in dry weight of the pile due to the microbial degradation of the organic matter for their calculation, which may be based on mass balance.

Response 16: please see Response 5.

There is no discussion of pelletizing, although it was the objective of the paper. The authors should re-consider their objectives and title of the paper.

Response 17: as above mentioned (please see Response 1), in this work we didn't studied the pelletization process. However the title "Composting as a solution to make pig manure suitable for pelletizing: environmental impact of the process" has been replaced with "Evaluation of two composting strategies for making pig slurry solid fraction suitable for pelletizing" according to Reviewer #2

Any conclusion related to pelletizing should be removed as that has not been discussed or studied

Response 18: in the Conclusion section no sentences related to pelletizing process have been reported

References: AOAC (1990) is missing from the reference list. References for manure composting need to be updated.

Response 19: Reference "AOAC,1990" has been now added in the reference list.

The quality of all figures should be improved.

The styles of the lines and symbols for the different piles in Figs 1 and 3 are too similar for identifying the different systems.

Use dots for decimal points.

In Figure 3, the title of the Y axis should follow the style of the other figures, e.g. CO_2 (g m^{-3} h^{-1}).

Insert error bars in data of Figure 2.

Response 20: done

Reduce decimals for most standard errors.

Response 21: done

Consiglio Nazionale delle Ricerche
IMAMOTER
Istituto per le Macchine Agricole e Movimento Terra

Editorial Office of Atmospheric Pollution Research

Title: *EVALUATION OF TWO COMPOSTING STRATEGIES FOR MAKING PIG SLURRY SOLID FRACTION SUITABLE FOR PELLETIZING*

20 July 2015

Dear Editors,

Please, find enclosed electronic version of the manuscript entitled "Evaluation of two composting strategies for making pig slurry solid fraction suitable for pelletizing" by PAMPURO Niccolò, DINUCCIO Elio, BALSARI Paolo and CAVALLO Eugenio, to be considered for publication in *Atmospheric Pollution Research*.

By signing this covering letter, I accept the conditions laid down in the Directions to Contributors:

- the submission represents original work that has not been published previously,
- it is not currently being considered by another journal,
- if accepted for the *Atmospheric Pollution Research* it will not be published elsewhere in the same form, in English or in any other language,
- each author has seen and approved the contents of the submitted manuscript.

As suggested in "Guide for Authors" I report the significance of results.

Composting of pig slurry solid fraction for pellet production is nowadays getting a popular manure management practice in most European countries. However, to our knowledge no data on the environmental impact of this process is available. This study was carried out to narrow the gap in knowledge by assessing, in a pilot scale experiment, NH_3 and GHG (CO_2, CH_4 and N_2O) emissions during composting of pig solid manure in windrows. Two composting strategies, turned and not turned, were investigated.

Results from this study provide useful information to be incorporated into decision tools to meet the needs of policy makers, advisors and farmers. In addition, data obtained in this study can be used for the compilation of national inventories of NH_3 and GHG emissions from the livestock sector.

If you need anything else, please do not hesitate to contact me.

Looking forward to hearing from you.

Yours faithfully,

Niccolò Pampuro.