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Composition, potential emissions and agricultural value of pig slurry from Spanish commercial farms

W. Antezana · C. De Blas · P. García-Rebollar · C. Rodríguez · A. Beccaccia · P. Ferrer · A. Cerisuelo · V. Moset · F. Estellés · M. Cambra-López · S. Calvet

Abstract Pig slurry is a valuable fertilizer for crop production but at the same time its management may pose environmental risks. Slurry samples were collected from 77 commercial farms of four animal categories (gestating and lactating sows, nursery piglets and growing pigs) and analyzed for macronutrients, micronutrients, heavy metals and volatile fatty acids. Emissions of ammonia (NH_3) and biochemical methane potential (BMP) were quantified. Slurry electrical conductivity, pH, dry matter content and ash content were also determined. Data analysis included an analysis of correlations among variables, the development of prediction models for gaseous emissions and the analysis of nutritional content of slurries for crop production. Descriptive information is provided in this work and shows a wide range of variability in all studied variables. Animal category affected some physico-chemical parameters, probably as a consequence of different slurry management and use of cleaning water. Slurries from gestating sows and growing pigs tended to be more concentrated in nutrients, whereas the slurry from lactating sows and nursery piglets tended to be more diluted. Relevant relationships were found among slurry characteristics expressed in fresh basis and gas emissions. Predictive models using on-farm measurable parameters were obtained for NH_3 ($R^2 = 0.51$) and CH_4

($R^2 = 0.76$), which suggests that BMP may be estimated in commercial farms from easily determined slurry characteristics. Finally, slurry nutrient composition was highly variable. Therefore, complete analyses of slurries should be performed for an effective and environmental friendly land application.

Keywords Ammonia emission · Methane emission · Fertilizer value · Prediction model · Slurry characterization

Abbreviations

BMP	Biochemical methane potential
CH_4	Methane
DM	Dry matter
EC	Electrical conductivity
CV	Coefficient of variation
NH_3	Ammonia
OM	Organic matter
SV	Volatile solids
TKN	Total Kjeldahl nitrogen
TAN	Total ammonia nitrogen
VFA	Volatile fatty acids

Introduction

At present, there is an increasing concern about the environmental impacts associated with intensive and concentrated livestock production. This is especially the case for pig production which currently has a global population of over 1 billion heads, mainly concentrated in intensive producing areas. Spain is the second largest pig producer in Europe and the sixth in the world, with about 26 million animal places (EUROSTAT 2015). Slurry management is influencing emissions of atmospheric pollutants such as ammonia (NH_3), nitrous oxide (N_2O) and methane (CH_4) (Hristov et al. 2013; Viguria et al. 2015) and it is also linked to potentially harmful effects for soil and water (Aarnink and Verstegen 2007).

Nevertheless, slurry should be considered a valuable resource as fertilizer because it contains significant amounts of most nutrients required by crops (Villar et al. 2004; Penha et al. 2015) plus organic substance which is important for soil fertility. The positive or negative effects of land application of slurry may depend on slurry characteristics (Villamar et al. 2013), dosing (Iguácel et al. 2011) or environmental conditions during application and hereafter (Webb et al. 2014). Moreover, pig slurry is used as a renewable source of energy due to its potential to produce CH_4 under anaerobic conditions (Ferrer et al. 2014; Zhang et al. 2014).

The composition of pig slurry from commercial farms is highly variable due to differences among housing systems, feed composition, climate or farm management (Beccaccia et al. 2015; Moral et al. 2005b, 2008; Sánchez and González 2005). In recent years, several studies have been published either characterizing physicochemical composition of slurries from commercial pig farms (Suresh et al. 2009; Parera i Pous et al. 2010), or evaluating the potential to emit NH_3 and greenhouse gases (GHG) form slurries obtained under experimental conditions (Antezana et al. 2015; Galassi et al. 2010; Hernández et al. 2013; Jarret et al. 2012 among others). However, there is scarce information on the potential emissions of GHG and NH_3 of slurries from commercial pig farms.

Understanding the relationships between physicochemical properties of slurry can provide a basis for estimating the fertilizer value of slurry, thus facilitating its more efficient use in agriculture and reducing the potential risks to the environment (Scotford et al. 1998; Díez et al. 2006; Thygesen et al. 2012).

It is therefore necessary to evaluate in detail the physicochemical characteristics of slurries in commercial farms, to analyze their relationships with the emission of CH_4 and NH_3 and to examine the potential fertilizer value of slurries. A previous study (Beccaccia et al. 2015) analyzed relationships among different feed and slurry characteristics and potential gaseous emissions. The aim of this study was to analyze in depth the variability in mineral composition and gas emission of pig slurry samples from commercial pig farms, to predict potential NH_3 emissions and the biochemical methane potential (BMP) in commercial farms and to explore limitations when using commercial slurries as fertilizers.

Materials and methods

Description of the selected farms and sample collection

A total of 77 samples of pig slurry from farms located in the Center and East of Spain were analyzed in order to account for potential variations in composition and emissions. A detailed description of the survey protocol and analysis can be found in Beccaccia et al. (2015). Samples were classified according to the animal category: 15 samples from gestating sows, 14 from lactating sows, 14 from nursery piglets and 34 from growing pigs. Table 1 summarizes the most relevant characteristics of the surveyed farms.

Sampling was designed to be representative of the slurry extracted from the pits under commercial conditions. A minimum of five aliquots (2-L) were taken during the pit discharge at equidistant time intervals. Composite samples were then thoroughly mixed in a 15 L container and subsamples were conditioned for the corresponding analyses. All samples were kept in sealed containers at 5 °C until laboratory processing, which was made within 24 h.

Physicochemical analysis

All pig slurry samples were analyzed to determine dry matter (DM), organic matter (OM) and total Kjeldahl nitrogen (TKN) according to APHA (2005). Also, pH, electric conductivity (EC), total ammonia nitrogen (TAN) (4500 NH_3-B and 4500 NH_3-C procedures) (APHA 2005), and volatile fatty acids (VFA) (Jouany 1982) were determined. Volatile fatty acids were analyzed using a Focus Gas Chromatograph (Thermo, Milan, Italy) equipped with a split/splitless injector and a flame ionization detector with the addition of an internal standard (4-metil valeric).

Macronutrients, micronutrients and heavy metal concentrations (P, K, Ca, Mg, S, Al, B, Cd, Co, Cr, Cu, Fe, Li, Mn, Mo, Na, Ni, Pb and Zn) were determined after acid digestion by inductive-optical coupled plasma spectrophotometry (ICP-OES, ICAP 6500 Duo, Thermo Scientific, Walthamm, MA, USA).

Ammonia emissions

Ammonia emissions assays were performed with an ammonia trap system similar to that described by Ndegwa et al. (2009). Slurry subsamples of 0.6 kg with a volume: surface ratio of 0.0952 m^3/m^2 were placed in duplicate in 1-L closed containers maintained at 25 °C in a thermostatic water bath (Selecta, Spain). Containers were connected to an air pump which extracted air at a constant airflow rate of 1.2 L min^{-1}. During 11 consecutive days, the air was forced to pass through two absorption flasks

Table 1 Main operation and installation characteristics of the farms surveyed

	Gestating sows	Lactating sows	Nursery piglets	Growing pigs
Number of farms	15	14	14	34
Animal places per farm (average)	770	162	2319	3132
Type of housing	Group	Individual	Group	Group
Feeding type	Dry	Dry (71 %)	Dry	Dry
		Wet (29 %)		
Feed restriction	Yes	No (79 %)	No	No
		Yes (21 %)		
Slurry accumulation				
<1 month	21 %	93 %	50 %	41 %
1–3 months	7 %	7 %	50 %	24 %
>3 months	71 %	0 %	0 %	35 %
Slat				
Partial	64 %	21 %	21 %	41 %
Total	36 %	79 %	79 %	59 %
Ventilation type				
Natural	86 %	57 %	43 %	78 %
Mechanical	14 %	43 %	57 %	16 %

(impingers) in serial containing 100 mL of sulphuric acid 0.1 N each. The acid solution was replaced daily during the first 5 days, and every 48 h until the end of the assay (day 11). The NH_3 trapped in the impingers was quantified following 4500 NH3-D procedure (APHA 2005) using a detection electrode (Orion High Performance NH_3 Electrode, model 9512HPBNWP, Thermo Scientific, USA).

Biochemical methane potential (BMP)

The BMP was determined in a batch assay using 120 mL bottles following the methodology described by Angelidaki et al. (2009). Inoculum from a mesophilic pig slurry anaerobic digester reactor was used. The inoculum was pre-incubated during 15 days at 35 °C in order to deplete the residual biodegradable organic material. Pig slurry and inoculum were mixed to obtain an inoculum to substrate ratio of 1 in OM basis. Each sample was tested out by triplicate. Additionally, three blank bottles containing degasified inoculum were included in the measurements in order to determine the inoculum endogenous CH_4 production. The endogenous CH_4 production from the inoculum was subtracted from the CH_4 produced by the pig slurry on each biogas sampling day. After filling, each bottle was sealed with butyl rubber stoppers and aluminum crimps and the headspace was flushed with pure N_2 for 2 min. Bottles were then incubated at 35° ± 1 °C for 100 days. During incubation, biogas volume in each bottle was regularly monitored (from 1 to 10 days depending on biogas production) by pressure measurement of the headspace using a manometer (Delta Ohm, HD 9220, Italy). Methane concentration in the biogas was further analyzed using a Focus Gas Chromatograph (Thermo, Milan, Italy) equipped with a split/splitless injector and a flame ionization detector. Finally, the BMP was expressed per volume of slurry considering the OM content.

Statistical analyses

All slurry components were expressed and analyzed in wet basis. The descriptive statistical analysis of the results of the variables in this study was made by animal category through PROC MEANS of SAS Institute (2008). The effect of the animal type in the studied variables was analyzed using PROC GLM and PROC T TEST of SAS Institute (2008). The Correlation analysis among physicochemical slurry characteristics, as well as NH_3 and BMP, was done using PROC CORR of SAS Institute (2008). A multiple regression analysis was also conducted with a stepwise variable selection process using the PROC REG of SAS Institute (2008) to establish prediction models of potential emissions from easily measurable slurry characteristics (i.e. EC, pH and DM).

From the results of the physicochemical characterization of slurry, potential scenarios of nutrient loads associated to land application were obtained. These scenarios corresponded to limitations of organic manure application according to international regulations, in particular the European Union Directive 91/676/CE which specifies that the livestock manure applied to land each year shall not exceed the amount containing 170 kg N ha^{-1}.

Results and discussion

Physicochemical slurry characterization

Summary statistics for the physicochemical characterization of slurry for the different animal categories are presented in Table 2. As shown in this table, slurry characteristics varied considerably among and within animal categories. Only for some characteristics the category of animal was found to have a statistically significant effect. Correlations among the main physicochemical characteristics are shown in Table 3.

The main results of this study are in agreement with those reported for pig slurries from commercial pig farms in Spain (Moral et al. 2005a, b; Parera i Pous et al. 2010; Sánchez and González 2005, among others) and other countries (Abubaker et al. 2015; Martínez-Suller et al. 2008; Olusegun 2014; Suresh et al. 2009; Villamar et al. 2012). The EC in pig slurry ranged from 2.65 to 53.46 mS cm^{-1}, and was higher for growing pigs and gestating sows than for lactating sows and nursery piglets ($p < 0.05$). This difference may be attributed to the housing system and management practices such as slurry removal frequency and use of cleaning water. In fact, animal categories showing higher EC are those in which slurries tend to be stored for a longer time in slurry pits (see Table 1), thus enhancing drying and mineralization of slurry. On the contrary, lactating sows and weaners are those with

Table 2 Physicochemical characterization of pig slurries by different animal categories

Parameter	All samples (n = 77)		Growing pigs (n = 34)				Gestating sows (n = 15)			
	Mean	CV	Mean	Min	Max	CV	Mean	Min	Max	CV
Physicochemical characteristics										
Electrical conductivity (mS cm^{-1})	21.8	53.1	25.7a	6.59	53.5	52.1	21.6a	10.15	44.6	37.2
pH	7.52	5.50	7.39a	6.41	8.05	5.70	7.78b	7.54	8.03	1.8
Dry matter (kg m^{-3})	49.3	78.9	57.09	7.50	176.7	71.7	56.05	4.9	155.4	92.4
Organic matter (kg m^{-3})	36.7	82.8	43.51	4.50	145.1	75.8	41.1	2.8	120.5	94.9
Ash (kg m^{-3})	12.5	70.7	13.57	3.00	34.6	62.2	14.94	2.12	45.6	87.7
Macronutrients										
N (kg m^{-3})	4.79	64.5	6.01a	1.43	15.1	60.0	4.36a	1.62	9.09	53.9
P (kg m^{-3})	0.860	90.9	0.831	0.104	2.09	63.3	1.31	0.060	4.58	105.9
K (kg m^{-3})	1.66	64.3	2.05a	0.504	4.62	60.5	1.53a	0.261	3.81	59.2
Ca (kg m^{-3})	1.23	95.9	1.13a	0.227	3.14	71.9	1.97b	0.115	6.45	107.9
Mg (kg m^{-3})	0.560	84.1	0.59	0.081	1.39	61.8	0.738	0.034	2.66	107.0
S (kg m^{-3})	0.300	74.7	0.34	0.056	0.953	71.5	0.310	0.043	0.849	86.7
Micronutrients and heavy metals										
Al (g m^{-3})	43.3	85.2	41.57	3.20	115.9	78.1	47.9	1.44	152.9	99.5
B (g m^{-3})	1.84	69.5	2.13	0.483	6.48	70.4	1.55	0.185	3.73	73.0
Cd (g m^{-3})	0.013	88.3	0.011	0.002	0.034	78.8	0.015	0.000	0.049	103.8
Co (g m^{-3})	0.686	317.2	0.507	0.014	5.70	208	0.840	0.018	8.9	268.2
Cr (g m^{-3})	0.602	112.0	0.61	0.031	1.95	87.1	0.880	0.018	4.36	133.1
Cu (g m^{-3})	12.7	126.3	10.4a	0.684	59.75	113	6.12a	0.236	17.6	92.2
Fe (g m^{-3})	85.0	93.1	73.78	5.95	189.6	64.1	90.52	2.92	302.2	101.7
Li (g m^{-3})	0.149	117.9	0.145	0.017	0.797	126	0.189	0.009	0.963	131.7
Mn (g m^{-3})	22.6	89.2	24.41	3.05	79.5	67.5	28.59	1.19	123.3	116.2
Mo (g m^{-3})	0.313	87.5	0.355	0.044	1.21	85.1	0.253	0.008	0.901	98.3
Na (kg m^{-3})	0.425	69.5	0.527a	0.108	1.41	68.2	0.382a	0.061	0.912	58.3
Ni (g m^{-3})	0.433	98.2	0.474	0.047	1.46	77.3	0.558	0.015	2.69	125.6
Pb (g m^{-3})	0.128	111.9	0.096	0.007	0.335	78.6	0.115	0.002	0.316	91.7
Zn (g m^{-3})	109.9	148.1	74.33a	3.98	442.8	123	45.92a	1.71	124.9	86.1
Other characteristics										
C (kg m^{-3})	20.2	81.3	24.5	2.52	77.0	74.9	21.06	1.35	56.9	92.1
TAN (kg m^{-3})	3.07	64.6	3.94a	0.673	10.3	59.3	2.87ab	1.29	4.68	37.5
Organic N (kg m^{-3})	1.67	87.9	2.03	0.176	7.87	87.1	1.62	0.141	5.1	91.9
VFA (mol m^{-3})	75.4	106.1	105.8a	0.867	329.7	87.7	37.4b	4.21	84.5	86.7
Acetic (mol m^{-3})	47.2	112.3	66.55a	0.618	238.4	93.7	28.8b	3.65	65.6	80.5
Propionic (mol m^{-3})	14.9	100.8	19.87a	0.114	69.4	82.2	5.85b	0.231	14.0	105.3
Butyric (mol m^{-3})	6.49	164.9	10.36a	0.014	47.4	128	0.750b	0.024	3.37	146.0
Ratio acetic/propionic	6.42	117.6	5.40	0.921	45.2	151	8.45	3.59	16.2	56.6
Ratio acetic/butyric	53.7	134.8	28.8a	1.85	118.5	122	121.1b	15.6	325.9	89.4
Ratio butyric/propionic	0.353	96.2	0.474	0.010	1.28	84	0.153	0.023	0.663	124.8
Ratio C/N	4.05	53.9	4.04	1.24	11.4	55.4	4.17	0.733	10.5	68.9
Ratio N/P	7.82	61.8	8.65	2.55	26.1	49	7.52	1.41	30.5	97.3
Ratio N/K	3.15	36.8	3.18	1.25	5.67	31.1	3.23	1.61	6.2	39.4

Table 2 continued

Parameter	All samples (n = 77)		Growing pigs (n = 34)				Gestating sows (n = 15)			
	Mean	CV	Mean	Min	Max	CV	Mean	Min	Max	CV
Potential gas emission										
Emission NH_3 ($g\ m^{-2}\ day^{-1}$)	10.0	4.77	11.6a	3.53	22.7	42	9.37a	5.51	20.0	41.2
Emission CH_4 ($m^3\ m^{-3}$)	9.73	118	14.4a	0.421	51.0	97.7	4.09b	0.224	10.8	103.5

Parameter	Lactating sows (n = 14)				Nursery piglets (n = 14)				*p* value*
	Mean	Min	Max	CV	Mean	Min	Max	CV	
Physicochemical characteristics									
Electrical conductivity ($mS\ cm^{-1}$)	15.5b	6.26	25.6	38.5	19.0b	2.65	46.6	61.5	0.030
pH	7.66c	7.05	8.16	3.60	7.43ac	6.34	7.92	7.50	0.009
Dry matter ($kg\ m^{-3}$)	35.6	8.6	75.8	63.1	36.83	5.70	97.3	67.2	0.172
Organic matter ($kg\ m^{-3}$)	25.9	4.9	55.4	66.3	26.74	3.60	72.4	70.7	0.152
Ash ($kg\ m^{-3}$)	9.72	3.72	20.4	56.3	10.09	1.88	24.9	59.9	0.256
Macronutrients									
N ($kg\ m^{-3}$)	3.37b	1.69	5.97	39.0	3.71b	0.720	11.7	74.4	0.014
P ($kg\ m^{-3}$)	0.704	0.107	1.86	80.7	0.623	0.111	1.59	68.3	0.076
K ($kg\ m^{-3}$)	1.07b	0.583	1.58	32.4	1.42a	0.216	3.85	65.7	0.017
Ca ($kg\ m^{-3}$)	1.01a	0.198	1.96	61.2	0.895a	0.149	2.22	66.7	0.047
Mg ($kg\ m^{-3}$)	0.477	0.056	1.28	80.6	0.386	0.082	0.856	63.2	0.193
S ($kg\ m^{-3}$)	0.212	0.076	0.47	63.1	0.313	0.061	0.856	69.8	0.390
Micronutrients and heavy metals									
Al ($g\ m^{-3}$)	53.5	5.59	147.2	82.9	32.7	2.02	88.1	75.9	0.479
B ($g\ m^{-3}$)	1.43	0.408	2.82	56.4	1.86	0.252	4.33	62.4	0.272
Cd ($g\ m^{-3}$)	0.013	0.001	0.039	96.1	0.016	0.001	0.043	76.0	0.520
Co ($g\ m^{-3}$)	1.37	0.019	16.3	314	0.273	0.023	1.16	129	0.543
Cr ($g\ m^{-3}$)	0.451	0.037	1.3	90.2	0.44	0.042	1.68	93.0	0.262
Cu ($g\ m^{-3}$)	5.3a	0.598	17.2	93.8	32.8b	3.33	80.6	70.7	0.000
Fe ($g\ m^{-3}$)	107.5	6.47	413.5	113	83.9	5.48	242.9	92.9	0.603
Li ($g\ m^{-3}$)	0.145	0.025	0.434	88.0	0.118	0.015	0.305	82.3	0.755
Mn ($g\ m^{-3}$)	15.8	2.12	43.4	82.7	18.3	2.2	47.9	76.4	0.280
Mo ($g\ m^{-3}$)	0.271	0.026	0.988	103	0.318	0.039	0.701	71.7	0.607
Na ($kg\ m^{-3}$)	0.304b	0.127	0.539	43.6	0.344c	0.062	0.826	70.0	0.047
Ni ($g\ m^{-3}$)	0.303	0.051	0.732	71.0	0.331	0.031	1.03	90.3	0.298
Pb ($g\ m^{-3}$)	0.181	0.009	0.691	105	0.167	0.017	0.893	134	0.187
Zn ($g\ m^{-3}$)	57.9a	5.12	189.4	101	316.8b	35.5	907.1	84.5	0.000
Other characteristics									
C ($kg\ m^{-3}$)	13.8	2.45	28.1	66.2	15.4	2.03	43.1	70.5	0.125
TAN ($kg\ m^{-3}$)	1.94c	0.878	3.38	36.5	2.32b	0.325	7.58	80.2	0.003
Organic N ($kg\ m^{-3}$)	1.22	0.213	2.4	58.3	1.32	0.335	4.44	79.7	0.241
VFA ($mol\ m^{-3}$)	30.9b	1.589	107.5	102.0	72.7a	9.77	250.0	107	0.019
Acetic ($mol\ m^{-3}$)	17.7b	1.45	31.7	64.0	40.3ab	1.83	165.2	135	0.038
Propionic ($mol\ m^{-3}$)	6.95c	0.057	36.9	161	18.4a	2.06	40.0	79.9	0.015
Butyric ($mol\ m^{-3}$)	2.30b	0.028	14.9	205	5.47ab	0.042	23.8	148	0.038
Ratio acetic/propionic	10.1	0.758	25.7	93.4	3.28	0.131	11.8	113	0.162

Table 2 continued

Parameter	Lactating sows (n = 14)				Nursery piglets (n = 14)				*p* value*
	Mean	Min	Max	CV	Mean	Min	Max	CV	
Ratio acetic/butyric	75.5b	1.87	293.7	114	32.0a	2.92	142.9	141	0.002
Ratio butyric/propionic	0.274	0.02	0.511	74.7	0.284	0.003	0.645	92.4	0.073
Ratio C/N	3.81	1.15	6.6	44.4	4.18	1.81	7.33	43	0.958
Ratio N/P	7.13	2.58	20.2	64.9	6.79	3.04	12.6	41.6	0.547
Ratio N/K	3.34	1.75	6.59	41.4	2.81	1.65	6.53	44.2	0.215
Potential gas emission									
Emission NH_3 (g m^{-2} day^{-1})	7.29b	3.32	12.8	34.6	8.43a	1.47	14.8	51.6	0.050
Emission CH_4 (m^3 m^{-3})	4.45b	0.223	10.9	101	7.33a	0.822	25.1	107	0.019

Means within the same row followed by different letters were significantly different

* ANOVA, test for comparing the animal type (growing pigs, gestating sows, lactating sows and nursery piglets)

shorter slurry accumulation time and tend to use higher volumes of cleaning water. Therefore, slurries from lactating sows and weaners tend to be more diluted, with lower DM contents. Differences in the concentration of protein and minerals in the diet of pigs among animal types (Moral et al. 2008; Beccaccia et al. 2015) may also be contributing to differences in EC. As shown in Table 3 and evidenced by previous studies (Provolo and Martínez-Suller 2007; Yagüe et al. 2012), both DM and EC are significantly correlated with most slurry characteristics. These relationships support the idea of dilution as a main factor affecting nutrient composition of slurries, and thus they have been used to propose prediction models for nutrients (Martínez-Suller et al. 2008). The pH ranged from 6.3 to 8.0 with low variation among animal categories. Slurry pH from gestating sows was on average 7.78 and it was higher than in slurries from other animal categories ($p < 0.01$). This value could probably be associated to lower concentrations of VFA (Table 2).

Regarding macronutrients (N, P, K, Ca, Mg and S) their concentration in the slurry tends to be higher for growing pigs and gestating sows, which is statistically significant in the case of N, K and Ca. In this regard, Sánchez and González (2005) and Moral et al. (2005a) also found higher N concentrations in slurries from growing pigs and gestating sows. It must be considered that nutrient excretion is a consequence of the inefficiency in their use by the animals and therefore it is affected by nutritional factors (Morazán et al. 2015; Bai et al. 2014; Patience et al. 2015), which may differ among animal categories.

A major part of the slurry nitrogen is in inorganic form, mainly as TAN, which represented 65 % of the total nitrogen. These values are similar to those obtained by previous studies (Sánchez and González 2005), which reported values of 57 % of TAN. As shown in Fig. 1, both N and TAN were positively correlated with EC ($R^2 = 0.65$ and 0.79, respectively), which supports the conclusions of Martínez-Suller et al. (2008) and Moral et al. (2005b) that these components can be predicted in practice.

In relation to the P content in the slurry, a wide variation was found among animal categories (ranging from 0.06 to 4.58 kg m^{-3}). The average content was 0.86 kg m^{-3}, but no statistical differences were found among animal categories. In the case of K, the slurry from lactating sows (1.07 kg m^{-3}) had a lower content than for the other categories ($p < 0.05$). Regarding the Ca content, the slurry from gestating sows (on average 1.97 kg m^{-3}) had a higher content than for the other categories ($p < 0.05$). Finally in the case of Mg the average content was 0.56 kg m^{-3}, but no statistical differences were found among animal categories. This is in accordance with previous studies (Moral et al. 2008; Sánchez and González 2005; Abubaker et al. 2015).

The content of micronutrients and heavy metals in the slurry was not statistically affected by animal category in the case of Al, B, Cd, Co, Cr, Fe, Li, Mn, Mo, Ni and Pb. However, the content of Cu, Zn and Na were affected by the animal category. Particularly, the content of Cu and Zn, which are commonly used as promotors, were highest for nursery piglets (32.8 and 316.8 g m^{-3} respectively, which is about 3 to 6 times

Table 3 Correlation matrix between the main pig slurry characteristics

Slurry characteristic	pH	EC	DM	Ash	TKN	TAN	OM	VFA	Acetic acid
EC	−0.069ns								
DM	−0.068ns	0.726***							
Ash	0.585***	−0.362***	−0.557***						
TKN	−0.176ns	0.816***	0.867***	−0.509***					
TAN	−0.134ns	0.888***	0.801***	−0.467***	0.960***				
OM	−0.097ns	0.729***	0.996***	−0.595***	0.872***	0.816***			
VFA	−0.523***	0.720***	0.489***	−0.544***	0.568***	0.656***	0.516***		
Acetic	−0.409**	0.774***	0.545***	−0.507***	0.599***	0.698***	0.569***	0.957***	
Propionic	−0.444**	0.581***	0.324*	−0.448***	0.447***	0.509***	0.340**	0.846***	0.708***
Butyric	−0.677***	0.461***	0.341**	−0.518***	0.404**	0.459***	0.376**	0.846***	0.741***
P	0.375**	−0.289*	−0.085ns	0.271*	−0.216ns	−0.310*	−0.131ns	−0.504***	−0.438***
K	0.243ns	−0.205ns	−0.554***	0.659***	−0.342**	−0.237ns	−0.552***	−0.182ns	−0.187ns
Ca	0.452***	−0.185ns	−0.078ns	0.490***	−0.161ns	−0.251ns	−0.127ns	−0.485***	−0.420***
C	−0.577***	0.416***	0.404**	−0.750***	0.475***	0.451***	0.426***	0.606***	0.541***
Ratio C/N	0.430***	−0.238ns	−0.014ns	0.424***	−0.135ns	−0.177ns	−0.05ns	−0.434***	−0.375**
Emission NH_3	0.176ns	0.543***	0.312*	−0.148ns	0.513***	0.652***	0.327*	0.362**	0.420***
BMP	−0.316*	0.798***	0.805***	−0.587***	0.896***	0.903***	0.836***	0.731***	0.737***

Slurry characteristic	Propionic acid	Butyric acid	P	K	Ca	C	Ratio C/N	Emission NH_3
EC								
DM								
Ash								
TKN								
TAN								
OM								
VFA								
Acetic								
Propionic								
Butyric	0.637***							
P	−0.456***	−0.503***						
K	−0.073ns	−0.204ns	−0.025ns					
Ca	−0.499***	−0.464***	0.600***	−0.085ns				
C	0.615***	0.484***	−0.408**	−0.373**	−0.470***			
Ratio C/N	−0.445***	−0.373**	0.383**	0.156ns	0.481***	−0.541***		
Emission NH_3	0.295*	0.162ns	−0.189ns	0.124ns	−0.220ns	0.137ns	0.045ns	
BMP	0.561***	0.581***	−0.189**	0.124*	−0.2196**	0.137***	−0.219ns	0.611***

EC electrical conductivity, *DM* dry matter, *TKN* total Kjeldahl nitrogen, *TAN* total ammonia nitrogen, *OM* organic matter, *VFA* volatile fatty acids, *P* phosphorus, *K* potassium, *Ca* calcium, *C* carbon, *NH_3* ammonia, *BMP* biochemical methane potential

*, **, *** Significant at $p < 0.05$; $p < 0.01$; $p < 0.001$, respectively; *ns* not significant

higher than other animal category; $p < 0.001$). On the contrary, the Na content was highest ($p < 0.05$) for growing animals (0.53 kg m^{-3}). The content of heavy metals expressed per dry matter basis is within the range reported by Nicholson et al. (1999). These authors reported average concentrations of Cu, Ni, Pb and Cd (351, 10.4, 2.48, 0.30 mg kg^{-1} DM, respectively), which is very similar to this study (297, 8.27,

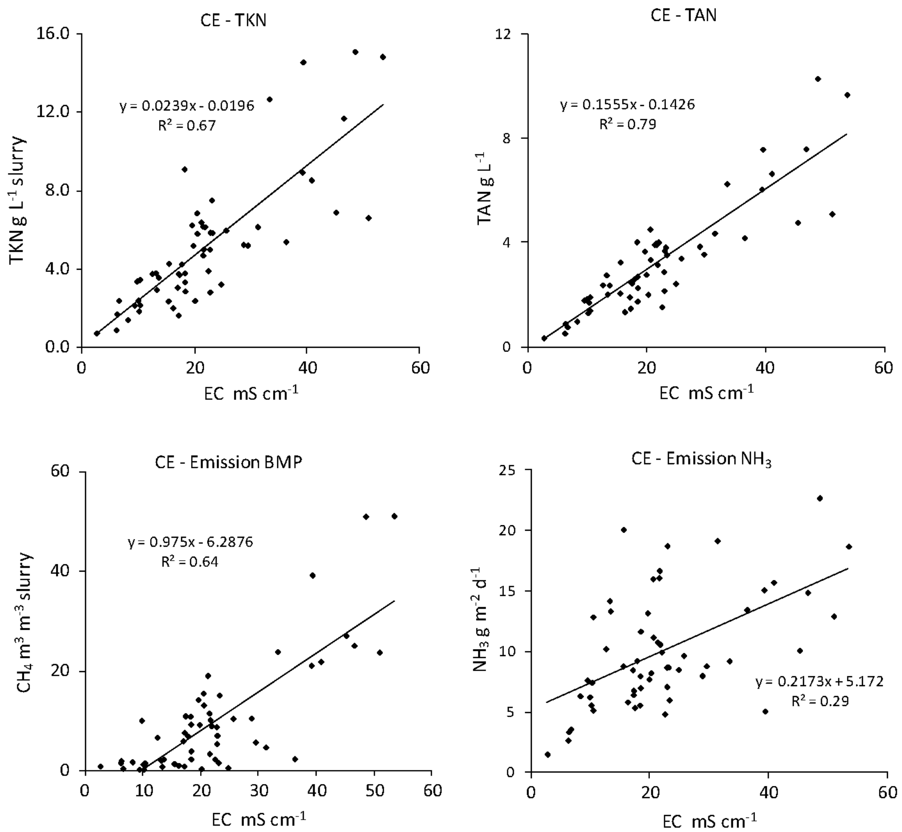

Fig. 1 Relationship between the electric conductivity (EC) and amount of total Kjeldahl nitrogen (TKN), total ammonia nitrogen (TAN), CH_4 emission and NH_3 emission in pig slurry samples (n = 77)

2.97 and 0.32 mg kg^{-1} DM, respectively). On the contrary, the concentration of Zn and Cr was from 3 to 5 times higher in our study (2540 vs. 575 mg kg^{-1} DM for Zn and 10.7 vs. 2.82 mg kg^{-1} DM for Cr, respectively).

Finally, the content in VFA in the slurry was higher for growing animals than for sows ($p = 0.019$). These results are coherent with those obtained by Conn et al. (2007), who found higher nutrient and VFA contents for finishing animals and attributed these differences to the dilution of slurry. Storage time and temperature have also been reported to reduce the VFA content (Popovic and Jensen 2012; Moset et al. 2012), while a dependency between animal nutrition and the VFA content of pig slurry was reported Aarnink and Verstegen (2007).

Gaseous emissions

The measured NH_3 emission of slurry was on average 10 g m^{-2} day^{-1}, ranging from 1.47 to 22.7 g m^{-2} day^{-1} (Table 2). The emissions were higher for slurries from growing pigs (11.6 g m^{-2} day^{-1}), gestating sows (9.37 g m^{-2} day^{-1}) and nursery piglets (8.43 g m^{-2} day^{-1}), compared to lactating sows (7.29 g m^{-2} day^{-1}) ($p = 0.05$). These results are apparently associated with the amount of TKN (r = 0.51) and TAN (r = 0.65) in slurries, which in turn is affected by the level of protein used in the diet (Beccaccia et al. 2015). As mentioned before, the TKN present in slurries is mainly in form of TAN (65 % in our slurry), which is the main source of NH_3 emission to the atmosphere. The content of TAN in pig slurries from growing pigs and gestating sows (3.94 and 2.87 kg m^3, respectively) were higher than for lactating sows and nursery piglets (1.94 and 2.32 kg m^3, respectively), which would indicate a greater availability for volatilization of NH_3 in slurry from growing pigs and gestating sows. A positive correlation was also found between NH_3 emissions and EC, but not with slurry pH (Table 3; Fig. 1). This finding is relevant because pH has been mentioned as an essential factor affecting NH_3 emissions (Snoek et al. 2014), but the results of this study do not reflect a direct effect of pH in a univariate analysis. Considering the variable nature of the samples from commercial farms used in this study, this suggests that the effect of pH may be partially confounded with the effect of TAN. Therefore, the relevance of these factors under commercial management should be further explored.

The BMP showed a wide variation, from 0.22 to 51 m^3 of CH_4 per m^3 of slurry (Table 2). The emissions of slurries from growing pigs and nursery piglets had a significantly higher BMP (14.4 and 7.33 $m^3$of CH_4 per m^3 of slurry respectively) than those from gestating and lactating sows (4.09 and 4.45 $m^3$of CH_4 per m^3 of slurry respectively). As indicated by Gopalan et al. (2013) and Beccaccia et al. (2015), changes in management practices and nutrition may be responsible for these differences between animal categories. The BMP was positively related with EC ($r = 0.798$; Fig. 1; Table 3) as well as acetic, propionic, butyric acids (Table 3; $r = 0.737$, 0.561 and 0.581, respectively). As indicated by Adekunle and Okolie (2015), these VFA are precursors of methanogenic activity and therefore may be potential indicators of BMP. Although accumulation of VFA may inhibit methanogenesis (Weiland 2010), in the conditions reported in this study, it seems that no inhibition was produced. The VFA content showed similar differences among animal categories as the BMP, and was higher for growing animals than for sows (Table 2). As indicated by Beccaccia et al. (2015), different animal categories are fed with different contents and types of fibers, which can influence the VFA contents in slurry.

The relationships described above can be used to predict potential emissions from slurry characteristics, as suggested by Yagüe et al. (2012) for other slurry physicochemical characteristics. Prediction equations for emission CH_4 and NH_3 were obtained, from easily measurable characteristics such as EC, pH and DM (Table 4). It can be observed that equations for predicting CH_4 emissions had higher coefficients of determination than those for NH_3 emissions (R^2 from 0.64 to 0.76 for CH_4 and from 0.30 to 0.51 for NH_3). However, the prediction equations improved when the models incorporated additional characteristics of the slurry (TKN, OM and TAN). Coefficients of determination including these variables reached 0.9 and 0.8 for CH_4 and NH_3, respectively. Other authors such as (Triolo et al. 2011) proposed prediction equations of BMP based on the content of different fiber fractions. However, the equations presented here can be of highest interest when estimating or comparing potential emissions per volume of slurry at farm level because easily measurable variables have been considered in the prediction models.

Potential fertilizer use

Considering the maximum permissible application rate of livestock manure to lanspecified in European regulations (170 kg N ha^{-1} $year^{-1}$), Table 5 shows nutrient inputs per hectare for land application using the slurries analyzed in this study. The volume of slurry necessary to achieve a supply of 170 kg N ha^{-1} was on average about 52 m^3 ha^{-1}, but was highly variable among farms (from 11 to 236 m^3 ha^{-1}) because of the variation in the concentration of N in the slurries. The corresponding application of OM ranged from 259 to 3520 kg ha^{-1}. As evidenced by previous studies (Fangueiro et al. 2012b; Hernández et al. 2007), the input of OM to soil through pig slurry is relatively low compared with other manure categories due to the relatively small content of organic carbon and the labile nature of the organic compounds. Therefore, according to these authors, the application of pig slurry does not significantly contribute to soil OM but it has been suggested that in the short term it can induce a reactivation of soil microbial activity.

The amount of mineral N incorporated as TAN also varied considerably among animal categories, with different implications. As indicated by Fangueiro et al. (2012a) and evidenced in this study, pig slurry contributes high proportions of TAN, and therefore it may be a valuable fertilizer for supplying N to plants. However, land application of livestock manures is associated to pollution risks related to NH_3 emissions (Krupa 2003) and N leaching (Mantovi et al. 2006). As mentioned before, due to the high TAN content found in commercial slurries, there is a high potential of NH_3 emission to the atmosphere. For this reason, abatement techniques during land application of pig slurry (e.g. surface application or injection) are of highest relevance (Webb et al. 2005).

The application of P and K varied by a factor 22 and 4, respectively among slurries. The variation in P load was particularly relevant for gestating sows (from 5.6 to 120.6 kg ha^{-1}). Agriculture uses about 80 % of

Table 4 Prediction equations for BMP ($L\ L^{-1}$) and NH_3 potential emissions ($g\ m^{-2}\ day^{-1}$) from pig slurry (n = 77)

No.	Equations	R^2	RSD
1	CH_4 emission = − 6.29 (±0.680) + 0.975 (±0.070) EC	0.637	4.6
2	CH_4 emission = 56.18 (±13.13) + 0.684 (±0.062) EC − 8.17 (±1.74) pH	0.658	4.3
3	CH_4 emission = 51.61 (±11.17) − 7.51 (±1.48) EC + 0.382 (±0.076)pH + 1.24 (±0.23) DM	0.757	3.5
4	CH_4 emission = 23.79 (±7.93) + 0.19 (±0.060) EC − 3.54 (±1.06) pH − 10.01 (±1.40) DM + 1.42 (±0.252) TKN + 13.84 (±1.79) OM	0.896	2.4
5	NH_3 emission = 5.17 (±0.919) + 0.217 (±0.037) EC	0.296	30.6
6	NH_3 emission = − 12.74 (±7.69) + 0.223 (±0.036) EC + 2.78 (±1.02) pH	0.431	26.9
7	NH_3 emission = − 18.48 (±7.12) + 0.172 (±0.056) EC + 2.99 (±0.939) pH − 0.603 (±0.162) DM	0.509	24.6
8	NH_3 emission = − 14.67 (±4.05) + 2.47 (±0.529) pH − 0.623 (±0.085) DM + 27.12 (±1.81) TAN	0.797	14.4

RSD residual standard deviation, *EC* electric conductivity mS cm^{-1}, *DM* dry matter, in percentage per L of slurry, *OM* organic matter, in percentage per L of slurry, *TKN* total Kjeldahl nitrogen, in percentage per L of slurry, *TAN* total ammonia nitrogen, in percentage per L of slurry

global P flows, and an efficient use of this nutrient is essential due to its non-renewable nature and the potential environmental impacts (Schröder et al. 2010). As described by Schoumans et al. (2014) and Dourmad and Jondreville (2007) adopting feeding strategies (use of highly digestible mineral P supplements, use of phytase and phase feeding) may considerably change the absorption and excretion balance of the animals. Therefore, different implementation of these techniques in commercial farms may be also contributing to the wide variation in the P content of pig slurry.

As indicated before, the concentration of macro nutrients expressed on a wet basis was higher for growing pigs and gestating sows than for nursery piglets and lactating sows. Furthermore, the ratio of major nutrients N:P:K in manure varied between animal categories (growing pigs 1:0.13:0.34; gestating sows 1:0.30:0.34; lactating sows 1:0.20:0.31; and nursery piglets 1:0.16:0.38). Variable ratios of these nutrients in pig slurry have been reported in the literature, particularly those corresponding to N:P ratios. Whereas some authors (Martínez-Suller et al. 2008; Parera i Pous et al. 2010) found relatively high contents of P in pig slurries (N:P ratios 1:0.51 and 1:0.59, respectively), other authors (Abubaker et al. 2015; 31 Sánchez and González 2005) detected slurries with relatively less P compared to N (N:P ratios 1:0.26, in both cases). In our study, the average N:P ratio was 1:0.18, which means relatively lower P compared to N than the ratios found in the literature for pig slurries. Differences in N:P ratios among studies could be attributed to factors affecting the efficiency in the use of P by the animals (e.g. nutritional factors), but could be also influenced by different slurry storage and sampling strategies among studies. The N:K ratios in the literature vary from 1:0.3 to 1:0.9 (Sánchez and González 2005; Martínez-Suller et al. 2008), which is consistent with the average ratio found in our study (1:0.3).

These ratios, however, do not necessarily correspond to the crop needs. For example, using the information provided by Van Duivenbooden et al. (1996) for cereals, it can be calculated that the required N:P:K ratio of fertilizer required is 1:0.39:1.34. According to our results, the average slurry would provide insufficient amounts of P and K. Although some particular samples were equilibrated in terms of N and P, most samples (particularly those from growing pigs) would be short of P. In all cases, for cereal production the K content of slurry would be insufficient to cover crop requirements. In this case, the lack of other nutrients in the slurry could be easily complemented with mineral fertilizers to obtain the optimal fertilization ratio. However, in practice fertilization ratios differ widely among crops, cropping systems and soil conditions (Penha et al. 2015) and as discussed here slurry composition is also very variable. Therefore, slurry characterization is required to avoid oversupply of nutrients and the corresponding environmental consequences, as well as to optimize the efficiency in the use of nutrients.

As regards heavy metals, loading rates were in accordance to those obtained by (Nicholson et al.

Table 5 Nutrient inputs per hectare to the soil by an application of pig slurry equivalent to 170 kg N ha^{-1}

Amount of components	Total simple (n:77)		Growing pigs (n:34)			Gestating sows (n:15)			Lactating sows (n:14)			Nursery piglets (n:14)		
	Mean	CV	Mean	Min	Max	Mean	Min	Max	Mean	Min	Max	Mean	Min	Max
Volumen (m^3)	51.9	72.2	40.0	11.3	119	50.9	18.7	105	58.1	28.5	101	75.8	14.6	236
Dry matter (Mg)	1.71	51.8	1.64	0.586	4.49	1.89	0.453	4.46	1.69	0.680	2.75	1.72	0.935	2.91
Organic matter (Mg)	1.26	56.1	1.23	0.362	3.47	1.38	0.259	3.52	1.21	0.387	2.13	1.24	0.522	2.23
Ash (Mg)	0.454	44.3	0.408	0.218	1.13	0.517	0.196	0.942	0.473	0.294	0.722	0.483	0.363	0.677
N (kg)	170	0.0	170	170	170	170	170	170	170	170	170	170	170	170
TAN (Kg)	112	22.7	114	54	152	119	75	157	107	69	152	106	72	136
Organic N (kg)	56.9	39.8	55.3	17.4	116	54.1	13.0	103	58.4	17.0	80.7	62.2	32.6	97.2
P (kg)	30.4	66.0	24.7	6.50	66.7	42.1	5.60	121	32.3	8.40	65.9	29.8	13.50	55.8
K (kg)	61.3	36.5	59.9	30.0	136	60.3	27.4	105	58.7	25.8	97.1	68.6	26.0	103
Ca (kg)	43.2	67.2	33.6	7.90	95.4	62.2	10.6	170	47.6	15.9	84.5	41.9	17.5	74.7
Mg (kg)	20.4	67.2	18.8	2.90	67.9	23.9	3.20	69.9	21.8	4.40	45.2	19.0	6.10	36.6
S (kg)	10.9	48.8	9.64	3.10	24.70	10.57	4.00	20.1	10.2	6.00	17.9	15.2	6.00	25.6
Al (kg)	1.62	84.1	1.32	0.300	5.80	1.54	0.100	3.40	2.49	0.400	6.00	1.56	0.200	4.00
B (g)	62.3	86.5	52.9	0.000	200	53.3	0.000	100	64.3	0.000	100	92.9	0.000	100
Cd (g)	0.492	80.4	0.326	0.100	1.00	0.480	0.000	1.10	0.571	0.100	1.40	0.829	0.100	1.80
Co (g)	27.6	345.7	13.9	0.500	147	45.1	1.70	560	59.0	1.50	622	10.7	2.30	39.1
Cr (g)	20.9	96.4	19.1	2.90	106	27.3	1.70	115	19.7	3.00	47.3	19.5	5.10	42.4
Cu (g)	509	132.3	299	79	1518	209	21.8	464	238	47.2	562	1611	148.6	3388
Fe (kg)	3.14	90.0	2.33	0.595	9.57	2.86	0.270	6.32	4.64	0.512	15.2	3.90	0.666	13.7
Li (g)	6.00	126.0	5.20	1.20	38.8	6.13	1.00	23.8	7.98	2.00	30.7	5.84	2.70	22.5
Mn (kg)	0.795	70.4	0.736	0.165	2.34	0.912	0.110	3.24	0.706	0.168	1.43	0.901	0.267	2.71
Mo (g)	11.2	72.2	10.4	2.70	35.7	8.7	0.80	23.7	11.9	2.00	32.4	15.0	4.80	38.4
Na (kg)	15.4	36.5	14.7	5.66	26.9	15.3	6.40	25.4	16.2	4.87	24.8	16.7	5.56	27.7
Ni (g)	15.1	78.7	14.4	4.90	52.2	18.0	1.40	70.8	14.2	4.10	24.9	14.7	6.70	36.7
Pb (g)	5.27	131.6	3.11	0.600	13.6	4.11	0.200	14.3	9.50	0.700	49.0	7.52	2.10	30.4
Zn (kg)	4.58	151.6	2.09	0.267	13.0	1.59	0.158	3.3	2.54	0.409	7.0	15.85	1.43	31.9
C (kg)	688	53.9	687	211	1940	709	125	1776	648	195	1122	711	307	1245

N nitrogen, *TAN* total ammonia nitrogen, *P* phosphorus, *K* potassium, *Ca* calcium, *Mg* magnesium, *S* sulfur, *Al* aluminum, *B* boron, *Cd* cadmium, *Co* cobalt, *Cr* chromium, *Cu* copper, *Fe* iron, *Li* lithium, *Mn* manganese, *Mo* molybdenum, *Na* sodium, *Ni* nickel, *Pb* lead, *Zn* zinc, *C* carbon

1999). Our study also evidences that the variability found in heavy metal concentrations involves variable loading rates of these elements when slurry is applied to soils. Therefore, although evident effects of heavy metals from organic substrates on crop production may not be noticeable (Diacono and Montemurro 2010), it is likely that heavy metal accumulates in areas where pig slurry is continuously applied for years (Nicholson et al. 1999). Regarding the potential effects of heavy metal accumulation, it must be considered that they also depend on their mobility. In fact, long term studies have demonstrated the accumulation of Cu, Zn and Pb in soils (Diacono and Montemurro 2010), but this did not affect soil productivity. According to these authors, soil properties such as cation exchange capacity, the presence of humic substances or the water and thermic regime are key factors affecting the mobility and thus the effects of heavy metals. The analysis of these properties is however beyond the scope of this study.

Conclusions

The composition of pig slurries from commercial farms has a wide range of variation in the main physicochemical characteristics. Some of these characteristics (pH, N, TAN, K, Ca, Cu, Na, Zn and VFA) were significantly affected by the animal category.

Emissions of NH_3 and CH_4 from pig slurries of commercial farms were highly related with the physicochemical composition, and were significantly affected by the animal categories. Potential emissions could be predicted using easily measurable slurry characteristics.

The direct application of pig slurries to cropland require characterization of the slurry, due to the wide variation found in its composition. Supplementation with nutrients or slurry treatment techniques may be necessary depending on slurry composition and crop needs.

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