Relationships among slurry characteristics and gaseous emissions at different types of commercial Spanish pig farms

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Abstract

This study aimed to analyse several factors of variation of slurry composition and to establish prediction equations for potential methane (CH₄) and ammonia (NH₃) emissions. Seventy-nine feed and slurry samples were collected at two seasons (summer and winter) from commercial pig farms sited at two Spanish regions (Centre and Mediterranean). Nursery, growing-fattening, gestating and lactating facilities were sampled. Feed and slurry composition were determined, and potential CH₄ and NH₃ emissions measured at laboratory. Feed nutrient contents were used as covariates in the analysis. Near infrared reflectance spectroscopy (NIRS) was evaluated as a predicting tool for slurry composition and potential gaseous emissions. A wide variability was found both in feed and slurry composition. Mediterranean farms had a higher pH (p<0.001) and ash (p=0.02) concentration than those located at the Centre of Spain. Also, type of farm affected ether extract content of the slurry (p=0.02), with highest values obtained for the youngest animal facilities. Results suggested a buffer effect of dietary fibre on slurry pH and a direct relationship (p<0.05) with fibre constituents of manure. Dietary protein content did not affect slurry nitrogen content but decreased (p=0.003) total and volatile solids concentration. Prediction models of potential NH₃ emissions (R²=0.89) and CH₄ yield (R²=0.61) were obtained from slurry composition. Predictions from NIRS showed a high accuracy for most slurry constituents (R²>0.90) and similar accuracy of prediction of potential NH₃ and CH₄ emissions (R²=0.84 and 0.68, respectively) to models using slurry characteristics, which can be of interest to estimate emissions from commercial farms and establish mitigation strategies or optimize biogas production.

Introduction

In the past, animal manure was regarded as a scarce and valuable source of plant nutrients to maintain soil fertility. However, at present there is an increasing concern about the impact of high levels of manure fertilization in different parts of the European Union, including some Spanish regions, where pig production is highly concentrated. According to the FAO Corporate Statistical Database (Faostat, 2014), world’s pig population has risen to almost 1 billion heads in 2012, and Spain is the sixth world pig producer. In this context, agriculture still plays an essential role in recycling manure nutrients, but also new uses of manure have been developed in recent years (e.g. biogas production).
Intensive livestock production constitutes an important source of emissions of ammonia (NH₃) and greenhouse gases (GHG) such as methane (CH₄) and nitrous oxide (N₂O) and, particularly in high producing areas, slurry management has been associated to nitrate contamination of ground and surface waters (Tamminga, 2003). In the European Union (EU-27), it is estimated that livestock contributed in 2012 to about 70% of NH₃ emissions to the atmosphere and pig production contributes to about 15% (EEA, 2014a). The management of livestock manure contributed to about 19% of total CH₄ emission, whereas slurry management in pig production emitted about 5.4% of total EU CH₄ emissions (EEA, 2014b).

It is widely recognized that there is a link between pig slurry composition and gas emission (Møller et al., 2004a; Dinuccio et al., 2008). Therefore, understanding the factors of variation of slurry composition under commercial conditions is essential to predict and control these emissions. However, it has also been reported that pig slurry composition in commercial farms is very heterogeneous and depends on multiple and interacting factors including the animal itself (breed and physiological status), feed composition and consumption, the housing system, manure management practices or environmental conditions (Sánchez & González, 2005; Conn et al., 2007; Moral et al., 2008; Martínez-Suller et al., 2010; Álvarez-Rodríguez et al., 2013).

Experimentally, it has been widely evidenced that nutritional strategies are effective to originate changes in the digestive performance of pigs and therefore influence the composition of excreta and thus the emissions of pollutant gases. The reduction of protein content of feeds affects directly nitrogen excretion and reduces NH₃ emissions, whereas slurry management in pig production affects the composition of excreta and thus the emissions of pollutant gases. The reduction of protein content of feeds has been reported to cause a shift in excreta from urinary to faecal nitrogen (Galassi et al., 2010; Halas et al., 2010) and reduce the pH of excreta (Kerr et al., 2006), thus reducing the emission of NH₃. On the contrary, increasing fermentable fibre content of pig feeds also enhances the emission of CH₄ from enteric origin (Jørgensen, 2007) and the CH₄ emission potential from slurry (Jarret et al., 2012).

A better characterization of the chemical components of pig slurry might improve the prediction of the associated gas emissions either from the animal house, the slurry storage or the soil after slurry application. Conventionally, slurry chemical composition is generally determined by using conventional wet chemical analysis performed at the laboratory which are expensive, time and labour costly, generate chemical wastes, and cannot be applied on-line. At farm level, however, rapid and low cost methods to predict slurry composition are necessary for an efficient use of slurry and as a consequence prediction methods have been developed during the last decade. These may be based on physico-chemical models (Chen et al., 2009; Yagüe et al., 2012) or the electrical properties (Bietresato & Sartori, 2013). Also, spectroscopic methods, as near infrared reflectance spectroscopy (NIRS) have found increasing use in the laboratory for low cost and rapid analysis, and offer a great potential for on-farm testing (Saëys et al., 2005). They have recently been applied for pig slurry analyses, and useful and accurate NIRS calibrations have been obtained for dry matter, ammonia N, total N and C (Malley et al., 2002; Saëys et al., 2005; Ye et al., 2005; Sørensen et al., 2007). Predicting methane potential emission using NIRS has also been recently a focus of interest to optimize anaerobic co-digestion processes (Doublet et al., 2013; Triolo et al., 2014). These calibrations need to be checked and updated periodically because of changes induced by variations in the slurry composition, but calibration maintenance, instrument validation, etc. could all be done on-line (Reeves, 2007).

As mentioned before, it is widely accepted that gaseous emissions in commercial farms are affected by a variety of dietary, animal, management and environmental factors. However, quantifying the relevance of factors affecting slurry composition and emissions at commercial level are currently topics of highest interest. Also, there is currently few published information on predicting potential NH₃ and CH₄ emissions from pig slurry at commercial farms using physico-chemical models or NIRS.

The objective of this work was to evaluate the relationships among slurry composition, gaseous emissions and several production factors (feed composition, season and location) in different types of commercial pig farms (nursery, growing-finishing, gestating and lactating sows). These relationships will be analysed throughout a multivariate analysis using a dataset of slurry samples covering a wide range of production conditions in commercial pig farms. Another objective was to establish predicting equations of potential gaseous emissions using physico-chemical models and NIRS.

Material and methods

Description of farm selection and sample collection

A survey protocol of commercial pig farms was established trying to cover the maximum variation in
commercial feeds, which could originate variability in the characteristics of slurries and in the potential NH₃ and CH₄ emissions. Representative feed and slurry samples were collected from 79 commercial pig farms (14 from either gestating, lactating and nursery piglets and 37 from growing-finishing animals) located at two regions in Spain (Centre and Mediterranean). Each farm was sampled once, either in winter or in summer. Farms were selected following three main criteria:

i) reflect the usual conditions of housing and manure management of intensive farming systems in Spain. In this way, dry feeding was generally provided in collective feeders (nursery and fattening pigs) and individual feeders (farrowing and gestation sows), most of farms used pelleted feed, access to feeding was restricted only for gestating sows, housing systems for fattening pigs and gestating sows were naturally ventilated, whereas both natural and mechanical ventilation was used for farrowing sows and nursery houses and proportion of slat was close to 100% in fattening, lactating and nursery farms, with lower and more variable values (from 25 to 100%) in gestating facilities.

ii) the slurry accumulated in the pits corresponds to the diets sampled when the survey was done and

iii) get a high variation among feed suppliers. Therefore, this survey protocol did not aim to be representative of the Spanish livestock sector, but to reflect potential variation in feeds, and as a consequence, on slurries and gaseous emissions.

The farms surveyed covered approximately a population of 11,000 sows, 35,000 nursery piglets and 60,000 growing-finishing pigs. The average storage time of slurry under the pits was about one month for lactating and gestating sows, as well as for nursery piglets and 51 days for growing-finishing pigs. The distribution of samples by zone, season and type of farm is shown in Table 1.

The samples were collected from March 2012 to February 2013, following a standardized protocol. Feed samples (1 kg) were taken from feeders or silos depending on their accessibility. Slurry samples were taken during pit discharge through a floor opening at the end of alleys, generally inside the barn. Sampling was made at regular time intervals by pooling a minimum of five aliquots (2-L) in a 15-L container and then subsampled into four 1-L plastic bottles for the different laboratory determinations, and stored at 4°C until analyzed. They were thoroughly mixed, subsampled into four 1-L plastic bottles for the different laboratory determinations.

### Feed and slurry chemical analysis

Slurry samples were maintained at 4°C and immediately analyzed at the arrival to laboratory (2-3 h after sampling) for pH (GLP21, Crison, Alella, Barcelona, Spain), electric conductivity (HI 98188-02, Hanna Instruments, Eibar, Spain), total solids (TS) and volatile solids (VS). The rest of analysis were made the day after, with samples maintained refrigerated at 4°C at the lab. Chemical analyses of samples were conducted in triplicate. The TS contents were determined after drying at 103°C for 24 h, and the VS after ignition in a muffle furnace (12-PR/300, Hobersal, Caldes de Montbui, Barcelona, Spain) at 550°C for 4 h. The total N and ammonia N concentrations were determined by steam distillation (APHA, 2005) using an automatic analyser (Pro Nitro A, J.P. Selecta S.A, Barcelona, Spain). Volatile fatty acids concentrations were determined by gas chromatography equipped with a flame ionization detector (HP 68050 series Hewlet Packard, USA) following the method described by Jouany (1982) with the addition of an internal standard (4-metil valeric).

The rest of slurry samples were dried at 60°C for 48 h, and feed and dried slurry samples were ground to pass through a 1-mm mesh screen (Cyclotec 1093 Sample Mill, Foss Electric A/S, Denmark). Dry matter (DM) and ash contents were carried out according to AOAC (2000) procedures 930.15 and 923.03, respectively. Concentration of neutral detergent fibre (NDF), acid detergent fibre (ADF) and acid detergent lignin (ADL) were determined sequentially by using the filter bag system (Ankom Technology, NY) according to Mertens (2002), AOAC (2000: procedure 973.187) and Van Soest et al. (1991), using heat stable amylase

| Type of animal         | Centre zone | Mediterranean zone | Total |
|------------------------|-------------|--------------------|-------|
|                        | Winter      | Summer             | Winter | Summer     |       |
| Gestating sows         | 3           | 4                  | 3      | 4          | 14    |
| Lactating sows         | 3           | 4                  | 3      | 4          | 14    |
| Nursery piglets        | 3           | 4                  | 3      | 4          | 14    |
| Growing-finishing      | 11          | 7                  | 11     | 8          | 37    |

Table 1. Number of feed and slurry samples classified by zone, season and type of animals.
Potential gaseous emissions

In vitro potential NH₃ emissions were determined by duplicate in laboratory following the methodology described by Portejoie et al. (2004). The samples (0.6 L each) were placed in 1 L closed chambers maintained at constant temperature (25°C) and connected to an air pump which extracted air from each chamber at an airflow rate of 1.2 L/min. During 15 consecutive days, the air was forced to pass through 2 absorption flasks (impingers) in serial containing 100 mL of 0.1 N H₂SO₄. The acid solution was changed every day during the experiment and analyzed for NH₃ content following 4500 NH₃-D procedure (APHA, 2005) using a detection electrode (Orion High Performance NH₃ Electrode, model 9512HPBNWP, Thermo Scientific, USA). The cumulative emission for each sample was calculated by adding the ammonium retained daily in the flasks during the experimental test.

Additionally, ultimate CH₄ yield (Bₒ) of each slurry sample was determined through biodegradability assays in 125 mL bottles during 100 days by using the methodology described by Vedrenne et al. (2007). These assays consisted in incubating different slurry substrates at mesophilic temperatures (35°C) in the presence of inoculum. Inoculum to substrate ratio was at unity or very close to unity on a VS basis (1:1). Each test on pig slurry was carried out in triplicate. Additionally, three blank bottles containing anaerobic sludge only were also used in order to determine the anaerobic sludge endogenous CH₄ production which was subtracted from the CH₄ produced by the pig slurry on each biogas sampling day. Bottles were then incubated at 35°C for 100 days. During incubation, biogas volume in each bottle was regularly monitored (intervals 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 analysed using a Focus Gas Chromatograph (Thermo, Milan, Italy) equipped with a split/splitless injector and a flame ionization detector.

According to the methodology of measuring in vitro emissions, these must not be considered as real emissions but as intrinsic properties of manure defining the potential to generate NH₃ and CH₄. Since all in vitro emissions are obtained in homogeneous environmental conditions, these potentials may be related to slurry characteristics. The effect of other variables (e.g. type of farm, season, and location) are therefore addressed in this study only in an indirect way: how these variables may affect slurry composition and thus their potential to emit NH₃ and CH₄.

NIRS determination

Feed and slurry samples were scanned using a Foss NIRSystem spectrophotometer (model 5000, Silver Spring, MD, USA) operating in reflectance mode and equipped with a sample transport device that allows samples be scanned while moving onto the same plane that equipment position. The spectra were acquired at 2 nm intervals over a wavelength range from 1,100 to 2,500 nm using the ISI NIRS 3 software ver. 3.11 (InfraSoft International, Port Matilda, PA, USA), and 32 co-added scans were averaged and collected by sample. Feed samples (undried and ground 1 mm) were scanned using a standard 1/4 sample cell and the equipment in a vertical position. The samples were mixed thoroughly using a homogenizer RW14 (Ika-Werke, Staufen, Germany) during 1 min; then, 80 mL were transferred to handmade polyethylene bags (60 × 230 mm) to a level of 10 cm from the bottom, and the upper part of the plastic bags was sealed after removing air space (Sørensen et al., 2007). The slurry sample bags were scanned by using a large sample cell (200 mm length by 4.7 mm width, and 23 mm depth) with the equipment placed on its back and the sample cell in a horizontal position. In this case, the particles were settled onto the face of cell and did not move out of the path of the light. Each slurry sample was measured in three independent subsamples by preparing three bags that were kept at 4°C and equilibrated at room temperature (15-20°C) before scanning. Each subsample was scanned twice mixing the contents to homogenize the sample between scanners, and averaged to provide one spectra per replicate. The average spectra of the three subsamples were used for chemometric analysis.

Statistical analysis

Slurry samples taken from independent facilities were the experimental unit for all the analyses. Descriptive analysis of the variables was performed through
PROC MEANS of SAS (SAS, 2008). Correlation analysis among dietary components and among slurry characteristics and feed composition was done using PROC CORR of SAS.

The prediction model of slurry composition and emissions included type of farm, season and location and their interactions as classified variables, as well as chemical constituents of feeds as linear covariates. PROC GLM of SAS was used to perform all of the analyses. A stepwise variable selection process was conducted using the PROC REG of SAS. To achieve the assumption of normality of the continuous variables analyzed, the Box & Cox (1964) transformation was used with PROC TRANSREG of SAS. The transformations used for each variable of feed composition and slurry characteristic are shown in Table 2. When significant differences of type of farm were detected, the Tukey test was used for mean comparisons.

Calibration models from NIRS for each constituent were performed using WINISI version 1.5 software, by modified partial least square regression based on cross-validation to avoid over-fitting of the equations. Prior to calibration, principal component analysis was performed to remove outliers with a standardized Mahalanobis distance H>3.0 (Shenk & Westerhaus, 1996) and no samples were marked as outliers. Different math pre-treatments of spectral data over three different segments (1,100 to 1,800, 1,200 to 2,400, and 1,100 to 2,500 nm) of the spectral range were tested, including none and three scatter correction techniques, standard normal variate and detrending, multiple, and inverse multiplicative scatter correction (Barnes et al., 1989) together with either or no first or second order derivatives, giving a total of 75 spectral models for each predicted parameter. The statistics used for selecting the best equations were the coefficient of multiple determinations \( R^2_{CV} \) and the standard error of cross-validation (SECV). The prediction accuracy for a model was based on the ratio of standard deviation (SD) of the reference data to the SECV, which should be at least three for an industrial application (Williams, 2001). The practical accuracy of NIRS calibrations developed was investigated by comparing the SECV to the standard laboratory errors. The repeatability of the predictions from the NIRS method was estimated from the variability of the values predicted in homogeneous analytical conditions from three subsamples.

**Results**

The average values of main chemical constituents of feeds and its variation (range and standard deviation) within the different types of farms studied are shown in Table 3. The results indicate an important variability among the samples analysed. The mean coefficients of variation for crude protein (CP) and NDF were 8.8 and 12.4%, respectively, and rose to 28-36% for NDICP, ADL and EE. The average values of slurry characteristics and its variability for the whole data set studied are presented in Table 4. The coefficients of variation (CV) of chemical components varied from 35-50% (for NDF, EE and total N) to more than 80% in the case of TS, VS, NDICP and ADL. The less variable characteristics were pH (CV=5.6%) and proportion of ammonia N on total N (CV=20%).

Correlation analyses were done: a) among feed constituents and b) between feed composition and some selected slurry characteristics (see results in Table 5). A negative correlation was observed between dietary CP and dietary fibre constituent contents. Dietary fibre components were significant and positively related among them and with ash and NDICP, which instead was little related with dietary CP level. Ether extract concentration in feeds was little related with any of the other components analysed. Otherwise, dietary concentrations of CP and NDICP were negatively associated to TS, VS and NDF, but positively with EE content of the slurry. Fibrous feed constituents were significantly and positively correlated with pH and NDF content in the pig manure and negatively with slurry EE concentration. Dietary EE and ash contents were little related with slurry characteristics, although the later was negatively associated to ammonia N and EE contents in the slurry. Otherwise, electric conductivity was highly correlated \( (p<0.001) \) with total and ammonia N in the slurry \( (r=0.803 \) and 0.884, respectively).
Table 3. Variability of feed composition within the different types of farms studied (% DM basis)

|                      | Ash | CP  | NDICP | NDF  | ADL  | EE   |
|----------------------|-----|-----|-------|------|------|------|
| **Gestation sows, n=14** |     |     |       |      |      |      |
| Mean                 | 6.25| 15.0| 2.37  | 22.6 | 2.28 | 4.93 |
| Min                  | 4.43| 12.6| 1.65  | 19.4 | 1.24 | 2.24 |
| Max                  | 7.99| 17.6| 5.15  | 29.3 | 3.77 | 7.57 |
| SD                   | 1.04| 1.48| 0.92  | 2.72 | 0.73 | 1.48 |
| **Lactating sows, n=14** |     |     |       |      |      |      |
| Mean                 | 6.54| 17.9| 2.26  | 19.0 | 1.79 | 5.85 |
| Min                  | 5.56| 15.7| 1.45  | 14.6 | 1.01 | 3.49 |
| Max                  | 7.52| 21.6| 4.14  | 24.5 | 2.70 | 8.01 |
| SD                   | 0.56| 1.54| 0.65  | 3.01 | 0.58 | 1.25 |
| **Nursery piglets, n=14** |     |     |       |      |      |      |
| Mean                 | 5.90| 19.0| 1.99  | 14.0 | 0.91 | 5.47 |
| Min                  | 5.01| 16.9| 1.29  | 11.3 | 0.39 | 2.26 |
| Max                  | 6.97| 21.5| 3.30  | 16.2 | 1.58 | 7.72 |
| SD                   | 0.49| 1.51| 0.56  | 1.48 | 0.37 | 1.87 |
| **Finishing-growing, n=37** |     |     |       |      |      |      |
| Mean                 | 5.17| 17.1| 2.01  | 16.7 | 1.31 | 5.47 |
| Min                  | 3.06| 13.7| 1.30  | 11.9 | 0.41 | 1.71 |
| Max                  | 6.58| 19.9| 2.53  | 20.5 | 2.51 | 9.16 |
| SD                   | 0.76| 1.52| 0.34  | 1.94 | 0.51 | 1.73 |

1CP=crude protein; NDICP=neutral detergent insoluble crude protein; NDF=neutral detergent fibre; ADL=acid detergent lignin; EE=ether extract

Table 4. Variability of slurry among the farms studied (n=79), % DM basis

|                     | pH  | Total solids | Ash  | Nitrogen | Ammonia-N | NDICP | NDF  | ADF  | ADL  | Ether extract |
|---------------------|-----|--------------|------|----------|-----------|-------|------|------|------|--------------|
| **Gestation sows, n=14** |     |              |      |          |           |       |      |      |      |              |
| Mean                | 7.77| 5.87         | 30.5 | 13.2     | 10.1      | 5.66  | 39.5 | 20.7 | 8.96 | 6.94         |
| Min                 | 7.54| 0.49         | 21.1 | 4.12     | 2.64      | 1.95  | 17.3 | 8.08 | 4.46 | 3.58         |
| Max                 | 8.03| 15.5         | 43.3 | 37.4     | 34.5      | 17.2  | 59.6 | 36.3 | 14.1 | 12.2         |
| SD                  | 0.14| 5.27         | 7.22 | 9.10     | 9.01      | 3.86  | 15.2 | 9.28 | 3.56 | 2.76         |
| **Lactating sows, n=14** |     |              |      |          |           |       |      |      |      |              |
| Mean                | 7.60| 3.58         | 29.2 | 10.9     | 7.38      | 6.17  | 38.7 | 19.6 | 8.88 | 9.32         |
| Min                 | 6.98| 0.86         | 21.7 | 6.02     | 3.11      | 1.75  | 24.4 | 12.3 | 5.43 | 4.03         |
| Max                 | 8.16| 7.58         | 43.3 | 24.5     | 20.9      | 18.8  | 48.9 | 27.5 | 16.1 | 15.5         |
| SD                  | 0.33| 2.23         | 6.82 | 5.58     | 5.73      | 4.66  | 7.81 | 4.82 | 2.66 | 3.56         |
| **Nursery piglets, n=14** |     |              |      |          |           |       |      |      |      |              |
| Mean                | 7.35| 3.73         | 29.7 | 11.3     | 7.53      | 5.98  | 29.5 | 12.6 | 5.12 | 13.0         |
| Min                 | 6.34| 0.57         | 22.4 | 5.62     | 2.52      | 0.23  | 3.79 | 1.07 | 0.39 | 6.28         |
| Max                 | 7.92| 9.73         | 42.1 | 23.0     | 19.3      | 16.4  | 48.0 | 21.2 | 10.2 | 18.8         |
| SD                  | 0.57| 2.42         | 5.91 | 4.68     | 4.44      | 4.93  | 13.2 | 6.52 | 2.73 | 4.31         |
| **Finishing-growing, n=37** |     |              |      |          |           |       |      |      |      |              |
| Mean                | 7.43| 5.38         | 26.7 | 12.8     | 8.92      | 4.24  | 34.5 | 16.6 | 6.78 | 10.8         |
| Min                 | 6.41| 0.75         | 15.5 | 4.18     | 1.32      | 0.56  | 5.25 | 2.24 | 0.59 | 2.93         |
| Max                 | 8.05| 17.7         | 44.3 | 27.9     | 24.1      | 14.9  | 56.6 | 36.2 | 18.3 | 24.9         |
| SD                  | 0.42| 4.08         | 6.87 | 5.16     | 4.63      | 3.22  | 12.4 | 7.53 | 3.57 | 4.03         |

NDICP=neutral detergent insoluble crude protein; NDF=neutral detergent fibre; ADF=acid detergent fibre; ADL=acid detergent lignin.

The effects of classified (type of farm, season and location) and of continuous (associated to feed composition) variables on slurry characteristics are shown in Table 6. Mean pH of manure was 7.50. It was not affected either by type of farm or season. An interaction between location and dietary NDF content was observed, as an increase of fibre concentration within type of farm increased linearly pH in farms placed in the
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Centre of Spain (from 7.08 to 7.77 in the natural scale between the extreme values of the range studied; \( p<0.001 \)), but not \( (p=0.522) \) in those located near the Mediterranean (Fig. 1). Total solids and VS concentrations in slurry were 4.85 and 3.61%, as average. They were not affected by either of the classified variables studied, but decreased linearly (by 84% in the natural scale, \( p=0.003 \)) when increasing dietary CP content. Ash slurry concentration on DM basis was affected by location; this variable was negatively transformed, so that values in Table 6 indicate that ash concentration was higher in the Mediterranean than in the Centre located farms (30.0 vs 26.1%, respectively, \( p=0.02 \)).

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### Table 5. Pearson correlation coefficients among some of the dietary and slurry characteristics studied

| Feed composition (% DM) | Ash | Crude protein | NDICP | NDF | ADF | ADL | Ether extract |
|-------------------------|-----|---------------|-------|-----|-----|-----|--------------|
| Ash                     | 1   | –             | –     | –   | –   | –   | –            |
| Crude protein           | –0.026 (0.818) | 1 | –     | –   | –   | –   | –            |
| NDICP                   | 0.260 (0.021)  | –0.107 (0.347) | 1     | –   | –   | –   | –            |
| NDF                     | 0.409 (<0.001) | –0.606 (<0.001) | 0.442 (<0.001) | 1   | –   | –   | –            |
| ADF                     | 0.485 (<0.001) | –0.450 (<0.001) | 0.377 (<0.001) | 0.864 (<0.001) | 1 | –   | –            |
| ADL                     | 0.596 (<0.001) | –0.443 (<0.001) | 0.489 (<0.001) | 0.810 (<0.001) | 0.852 (<0.001) | 1 | –            |
| Ether extract           | 0.022 (0.848)  | 0.180 (0.112)  | 0.129 (0.259) | 0.017 (0.879) | –0.129 (0.256) | –0.016 (0.888) | 1 |

### Slurry characteristics (% DM)

| pH       | TS (%)   | VS (%)  | NDF (%) | Total N | Ammonia N | Ether extract |
|----------|----------|---------|---------|---------|-----------|---------------|
| Feed composition (% DM) | 0.161 (0.156) | 0.049 (0.668) | 0.018 (0.877) | –0.070 (0.539) | –0.130 (0.252) | –0.228 (0.042) | –0.272 (0.015) |
| Crude protein | –0.054 (0.636) | –0.353 (0.001) | –0.357 (0.001) | –0.263 (0.019) | –0.027 (0.815) | –0.059 (0.607) | 0.256 (0.023) |
| NDICP    | –0.028 (0.807) | –0.233 (0.038) | –0.231 (0.040) | –0.207 (<0.067) | –0.083 (0.464) | –0.129 (0.258) | 0.140 (0.219) |
| NDF      | 0.257 (0.021) | 0.142 (0.213) | 0.134 (0.240) | 0.238 (0.035) | 0.092 (0.418) | 0.116 (0.308) | –0.471 (p<0.001) |
| ADF      | 0.343 (0.001) | 0.166 (0.142) | 0.147 (0.197) | 0.213 (0.058) | 0.066 (0.560) | 0.154 (0.176) | –0.516 (p<0.001) |
| ADL      | 0.300 (0.007) | 0.148 (0.193) | 0.133 (0.244) | 0.132 (0.246) | 0.138 (0.223) | 0.149 (0.190) | –0.490 (p<0.001) |
| Ether extract | 0.064 (0.576) | –0.169 (0.137) | –0.156 (0.171) | –0.124 (0.276) | 0.081 (0.473) | 0.071 (0.531) | 0.065 (0.564) |

1Values in parentheses indicate \( p \)-value; bold values indicate significant correlations. 2 NDICP=neutral detergent insoluble crude protein; NDF=neutral detergent fibre; ADF=acid detergent fibre; ADL=acid detergent lignin; TS=total solids; VS=volatile solids;

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Figure 1. Effect of the interaction between location and dietary NDF (neutral detergent fibre) content on \( \text{pH} \) of the slurry (Location=Centre ▪; Location=Mediterranean ▲)
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Table 6. Effect of the factors studied on slurry characteristics and potential emissions (% DM, except when indicated). Values in table are LS means. Means and regression coefficients of the continuous variables correspond to values transformed as indicated in Table 2.

| Item | Type of farm | Season | Location | SD | p |
|------|--------------|--------|----------|----|---|
| pH^1 | N  | 78.1 | 76.8 | 98.4 | 88.0 | 85.0 | 85.6 | 68.4 | 97.2 | 28.9 | 0.406 | 0.932 | <0.001 |
| Total solids (%)^2 | GF | 1.39 | 1.39 | 0.84 | 1.20 | 1.22 | 1.18 | 1.10 | 1.13 | 0.82 | 0.304 | 0.837 | 0.258 |
| Volatile solids (%)^2 | G  | 1.27 | 1.27 | 1.20 | 1.22 | 1.22 | 1.21 | 1.20 | 1.24 | 0.22 | 0.302 | 0.847 | 0.373 |
| Ash | L  | 0.188 | 0.197 | 0.180 | 0.193 | 0.191 | 0.188 | 0.194 | 0.185 | 0.021 | 0.639 | 0.259 | **0.021** |
| Total N | W  | 2.35 | 2.47 | 2.39 | 2.30 | 2.37 | 2.38 | 2.41 | 2.34 | 0.22 | 0.673 | 0.936 | 0.554 |
| Ammonia N | S  | 1.87 | 2.05 | 2.00 | 1.80 | 1.92 | 1.94 | 1.94 | 1.92 | 0.63 | 0.600 | 0.890 | 0.890 |
| Ammonia N (%N) | C  | 4.12 | 4.19 | 4.21 | 4.11 | 4.15 | 4.16 | 4.13 | 4.18 | 0.22 | 0.526 | 0.815 | 0.388 |
| NDF%^3 | M  | 29.7 | 34.3 | 39.7 | 38.8 | 36.9 | 34.4 | 37.2 | 34.1 | 12.5 | 0.481 | 0.416 | 0.275 |
| ADF^4 | 13.3 | 16.7 | 20.1 | 19.4 | 17.6 | 17.1 | 17.8 | 16.9 | 7.43 | 0.372 | 0.793 | 0.587 |
| ADL^5 | 2.21 | 2.52 | 2.89 | 2.94 | 2.65 | 2.63 | 2.65 | 2.63 | 0.64 | 0.125 | 0.857 | 0.857 |
| Ether extract^6 | 12.4 | 10.7 | 7.45 | 9.69 | 9.5 | 10.8 | 9.32 | 10.8 | 3.73 | **0.016** | 0.094 | 0.538 |
| VFA (mg/L)^7 | 18.8 | 16.1 | 7.47 | 12.5 | 12.9 | 14.6 | 15.0 | 12.5 | 5.19 | **0.002** | 0.266 | 0.068 |
| In vitro ammonia emissions (mg/L/day)^8 | 4.62 | 4.50 | 4.30 | 4.38 | 4.50 | 4.33 | 4.29 | 4.54 | 0.46 | 0.754 | 0.125 | **0.027** |
| Ultimate methane yield | 18.0 | 16.1 | 13.1 | 12.0 | 15.3 | 14.3 | 15.4 | 14.2 | 4.50 | **0.002** | 0.311 | 0.210 |

^1NDF=neutral detergent fibre; ADF=acid detergent fibre; ADL=acid detergent lignin; VFA=volatile fatty acids. ^2N=nursery; GF=growing-finishing; G=gestating; L=lactating; W=winter; S=summer; C=centre; M=mediterranean. ^3Effect of dietary NDF content and location: 99.9 ± 29.3 (Centre, GF; 97.8 ± 29.3 (M); 93.0 ± 29.3 (S); 92.8 ± 29.3 (W); 89.1 ± 29.3 (L)). ^4Linear effect of dietary CP: -1.51 ± 0.61; p=0.001. ^5Linear effect of dietary NDF: 12.0 ± 4.33; p=0.012. ^6Ether extract concentration on manure was not affected by the other classified variables studied. ^7In vitro ammonia emissions (mg/L/day)=-0.74 (±0.59) + 0.58 (±0.049) ln total N + 2.84·10^{-9} (± 9.57·10^{-10}) pH^9. ^8VFA decreased linearly with dietary CP content (from 10751 to 466 mg/L, p=0.002). Estimates of in vitro ammonia emissions from the slurry decreased in farms located in the Central region (p=0.027), but were not affected by the other classified variables studied; they also decreased with dietary ash and CP (by 55.8 and 77.4% in the range sampled in this study, p=0.001 and 0.015, respectively) and increased linearly with degree of lignification of NDF (by 137%, p=0.012), in the range of values studied. No significant effects were detected for any of the interactions among the main factors included in the model on any of the slurry characteristics studied. A better fit was obtained when expressing NH3 emissions on VS basis: In vitro ammonia emissions (mg/g VS)^11=-3.72 (±0.92) + 1.85 (±0.12) ln total N + 0.653 (±0.26) ln ammonia N (% total N) + 4.83·10^{-8} (± 1.50·10^{-10}) pH - 0.046 EE (R^2=0.888; RSD=0.378; n=79). In the case of potential CH4 yield, the fitted model was: B0 (mL/g VS)^12=-21.1 (±2.45) + 0.396 (±0.10) EE - 7.07 (±1.21) ADL/2 + 0.240 (±0.059) NDF (R^2=0.610; RSD=3.13; n=79).
According to these results, in vitro ammonia emission was not affected by type of farm, season or location, but increased with total N \((p<0.001)\) content, proportion of ammonia N on total N \((p<0.001)\) and pH \((p=0.007)\) of the slurry from 3.7 to 187; 25.1 to 48.5 and 29.2 to 54.4 mg/g VS and decreased with EE content \((p<0.001)\) from 52.8 to 20.1 mg/g VS, for the extreme values of the range studied. Potential methane production was neither affected by any of the classified variables, but increased \((p<0.001)\) with EE and NDF manure content (from 149 to 443 and 61.1 to 436 mg/g VS, respectively) and decreased with ADL concentration from 852 to 11.1 mg/g VS in the range of the samples studied.

Figures 2 and 3 represent, respectively, the changes of estimates of ammonia and methane production in the range of values studied, with respect to the slurry characteristics selected in the prediction models. Changes are expressed relatively taking as base=100 the average value of each of the independent variables considered, in the natural untransformed scale.

Calibration and cross validation statistics of prediction of laboratory analyses and ammonia and methane estimations from NIRS analysis are shown in Table 7. The coefficients of determination of calibration for chemical analyses were generally high, above 0.90 for DM, VS, total N and EE, being the lowest (from 0.70 to 0.85) those of ash, pH, and fibrous constituents; the coefficients of determination obtained for cross validation were similar but slightly lower. Coefficients of determination of cross validation for prediction of in vitro ammonia emissions and ultimate methane yield were respectively 0.836 and 0.682.

**Discussion**

**Effects of type of farm, season and location**

When considering in the model the effect of the covariates associated to dietary chemical composition, the classified variables studied had a limited influence on the slurry characteristics. Farm location had a significant effect on pH and ash of the manure, with the highest values observed in Mediterranean farms. Although feeding programs, genetic potential and systems of management are similar, there is a major difference in water quality between the two areas sampled. According to the Spanish Ministry of Health (http://sinac.msc.es/SinacV2/), the Mediterranean region has a...
In this study water characteristics were not analysed and therefore further studies in this sense would be necessary to confirm and characterize this effect.

The effect of type of farm on slurry traits was scarce, as the differences in average feed composition among animal categories were accounted by the model co-variates. Even so, a higher EE and VFA concentration and Bo were observed in the slurry samples taken from the youngest animals (nursery farms). Average feed EE content was similar among the different types of farms (see Table 3), but young pigs show a lower capacity of fat digestion (Soares & López-Bote, 2002) that would lead to a higher fat excretion and slurry concentration. Otherwise, EE is by far the nutrient with the highest capability of microbial fermentation and therefore a nutrient with high potential to generate methane in the slurry (Angelidaki & Sanders, 2004).

Season had no significant influence on any of the slurry components and this could explain the absence of seasonal effect on potential NH₃ and CH₄ emissions. Slurries were collected from indoor slurry pits, where climatic variations are attenuated, thus minimizing the seasonal effect. Other works (Møller et al., 2004b; Pereira et al., 2012) have shown lower methane and ammonia emissions at lower ambient temperatures, probably as a consequence of slower microbial and enzymatic reactions. This study, however, is not comparable since potential emissions are estimated under controlled, laboratory conditions. According to Angelidaki & Sanders (2004) temperature does not influence the ultimate biodegradability of a component, but may reduce the degradation rates. In addition, our results regarding CH₄ emissions are in accordance with Liu et al. (2014). In their review, these authors reported that temperature affected CH₄ emissions from lagoons, but was not a significant factor on housing CH₄ emissions from swine. Otherwise, apart from the intrinsic manure characteristics, NH₃ emissions are conditioned by ambient temperature and the convective mass transfer coefficient.

**Effects of dietary fibre concentration**

A higher fibre supply buffered the decrease of slurry pH in farms located at the Centre of Spain, where mean water and slurry pH were lower than in the Mediterranean area. This result might be explained by the buffering and cation exchange properties of some cell wall constituents (i.e. lignin, nitrogen and pectins; Van Soest, 1994), as an increase in dietary NDF concentration also increased slurry content of NDF, and more markedly those of ADL and ADF. These increments reflect the limited digestion efficiency of cell wall constituents in the pig, in inverse relation with its

![Figure 3. Effect of several slurry characteristics on the ultimate methane yield (Bₒ). Base 100=average value of each independent variable. ADL=acid detergent lignin; NDF=neutral detergent fibre.](image-url)
degree of lignification. In contrast with the current results, Canh et al. (1997) reported a reduction of pH of slurry in response to the dietary addition of digestible fibre in form of sugar beet pulp, and related it to a higher microbial fermentation in the hindgut. However, a lesser or none effect of dietary NDF on pH was observed in other studies when also supplementing diets with soluble fibre (Halas et al., 2010; Von Heimendahl et al., 2010), or with a mixture of soluble and insoluble fibre (Galassi et al., 2010).

In the same way, there is a general agreement in that inclusion of fermentable fibre, as sugar beet pulp or inulin, leads to a shift in the N excretion from faeces to urine (Aarnink & Verstegen, 2007), which is generally associated to an enhanced metabolic urea retention and excretion as microbial protein in the faeces (Kreuzer et al., 1999). This shift would imply in turn a decrease of ammonia emissions from the slurry with fibre supplementation but also a higher fermentation activity and methane losses, as observed by Montalvo et al. (2013). However, Galassi et al. (2010) did not observe a significant influence of supplementation of the diet with a mixture of 20% of wheat bran and 4% of sugar beet pulp on ammonia emissions and Triolo et al. (2011) reported a high negative correlation ($r=-0.952$; $p<0.001$) between lignin content in the manure VS and its biochemical methane potential. Moreover, Bindelle et al. (2009) demonstrated that the substitution of sugar beet pulp with a source of insoluble fibre, as oat hulls, decreased the synthesis of bacterial protein in the gut and the ratio faecal : urinary N to levels similar or lower than those reached with the standard non-supplemented diet; these results help to explain the positive relationship found in the current study between degree of lignification of dietary NDF and ammonia emissions from the slurry.

The proportion of soluble/insoluble fibre or the fermentability of the feeds sampled were not measured in the current study, but the average degree of lignification of the NDF was 8.48% (see Table 3), which is similar to those of wheat bran (8.83%) or oat hulls (8.98%), but clearly above to that found in sugar beet pulp (3.9% as average, according to FEDNA, 2010). The relatively lignified type of fibre more frequently used at present in Spanish commercial feeds for pigs would then explain the lack of effect of dietary fibre level on the ratio of ammonia to total N or the ammonia and methane emissions from the slurry.

**Effects of dietary N concentration**

The lack of influence of dietary protein concentration on ammonia and N content in the slurry differs from most of previous research that generally found a positive relationship between these variables (Canh et al., 1997, 1998; Hayes et al., 2004), although following great changes in dietary protein level (from 4 to 9 percentage units). However, Hernández et al. (2011) found little effect of dietary protein level on manure composition and ammonia emissions when working with commercial growing-finishing feeds and a narrower range of CP content (from 14 to 16%). In the same way, neither Portejoie et al. (2004) nor Kerr et al. (2006)
observed significant differences in ammonia or total N content in the slurry of growing pigs, when comparing diets containing 20 vs 16% or 14.5 vs 12.0% CP, respectively. This lack of effect might be related to the short range of variation used in commercial studies. In addition, Kerr et al. (2006) also suggest that a higher ammonia volatilization might occur in the slurry of pigs fed the greater CP diets before samples were taken. This effect may be particularly relevant for slurries stored for a long time (e.g. more than 3 weeks) in manure pits. In the current study, SD of CP content was around 1.5 within type of farms (Table 3), so that most of the diets were in a narrow range of three percentage units with respect to the mean.

Otherwise, an increase of dietary N concentration led to a linear decrease in TS content of the manure in the current study. Portejoie et al. (2004) showed in finishing pigs that lowering dietary protein level (from 20 to 12%) decreased DM concentration in the slurry (from 5.9 to 4.4%), because of a lower water consumption. The same trend (3.24 vs 2.51%) was observed by Kerr et al. (2006), although in this case the range of variation of protein content in the feed was shorter (from 14.5 to 12.0%) and the differences did not reach significant levels. The observed reduction of DM content in the manure when dietary CP concentration increased would also explain its negative effect on in vitro ammonia emissions per L of slurry, because of the parallel reduction of nutrient content (including ammonia and total N).

Effects of dietary ash concentration

The range of ash content in feeds DM within the different types of farms studied was from 2 to 3.5 percentage units, with SD ranging from 0.5 to 1.0 (Table 3). In commercial diets this variation is mostly related to Ca content (Sánchez & González, 2005), but also to the inclusion of clay in the feeds to improve pelleting characteristics. Some sources of Ca (as its anionic salts, sulphate or chloride) can reduce urine pH and then reduce emissions, but they are of little use in practical pig diets. Otherwise, undigested clay might increase ammonia absorptive properties of the slurry, which could help to explain the reduction of ammonia emissions observed in the current study with increasing ash concentrations in the manure.

Models of prediction of ammonia and methane potential emissions

The accuracy of the prediction of most of the organic constituents of pig slurry from NIRS was generally high which confirms the findings of other studies (Malley et al., 2002; Saeyts et al., 2005; Ye et al., 2005; Sørensen et al., 2007) on the usefulness of this methodology to predict main chemical components of fresh slurry and the biochemical CH4 potential in a range of organic substrates (Doublet et al., 2013; Triolo et al., 2014). The poor prediction for pH could be related to internal correlations to organic compounds giving no true correlations between pH and spectra absorbance peaks (Huang et al., 2007). The lowest $R^2$ vs values were obtained for fibrous components (as those of Van Soest fibre analysis) where analytical procedures tend to lead to a high analytical error. In addition, the prediction accuracy of chemical traits included as variables in chemical prediction models and those from NIRS help to explain the similar accuracy of the prediction of potential NH3 and CH4 emissions from the slurry composition or through NIRS methodology. The ratio of SD to SECV values in Table 7 was 2.46 for ammonia and 1.82 for methane potential emissions; these values are below the level of 3.0 that makes ideally the prediction “good” according to Williams & Sobering (1996), being only useful that of potential NH3 emissions for screening purposes, and emphasizes the need to enlarge the database used to increase variance of the reference data.

At commercial scale, it must be also considered that relating nutritional factors to gaseous emissions may not be straightforward. Manure composition and therefore gaseous emissions may be also affected by other factors of variation such as slurry management or temperature (Liu et al., 2013; Snoek et al., 2014) in the farm. The type of operation (e.g. sows, growing pigs, etc) has also been considered a relevant factor influencing manure composition as a consequence of manure dilution (Conn et al., 2007), but this effect may be confounded with dietary factors, since feed composition differs for animals in different physiological status. However, despite the influence of these variables, the prediction models of emissions in this study showed similar fitting to prediction models of other slurry components reported in the literature. For example, Yagüe et al. (2012) reported coefficients of determination ranging between 60 and 90% for physico-chemical models to predict most slurry constituents, whereas Triolo et al. (2014) found a coefficient of determination of 84% in predicting biochemical methane production of a wide variety of biomass samples.

In conclusion, the models developed in the current study in a wide range of practical conditions are useful to understand the factors behind changes in slurry characteristics and to predict NH3 and CH4 potential emissions. In addition, predictions from NIRS of gaseous emissions showed a similar accuracy to prediction models using slurry composition, and can be therefore
further explored to investigate potential pollution of livestock slurries, as well as for their use as biogas substrates.

Acknowledgments

We thank the Agencia Española de Cooperación Internacional para el Desarrollo (MAEC-AECID) and CAPES Foundation, Ministry of Education of Brazil, Brasilia -DF 70040-020, Brazil for research fellowships.

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