Acidification with sulfur of the separated solid fraction of raw and co-digested pig slurry: effect on greenhouse gas and ammonia emissions during storage

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Acidification with sulfur of the separated solid fraction of raw and co-digested pig slurry: effect on GHG and ammonia emissions during storage

F. GioelliA, E. DinuccioAD, D. CukA, L. RollèA, P. BalsariA

A Department of Agriculture, Forest and Food Sciences (DISAFA), Università degli Studi di Torino. Largo Paolo Braccini, 2 – 10095 Grugliasco (To)

D Corresponding author. Email: elio.dinuccio@unito.it - phone: +39 0116708718, fax: +39 0116708591

Keywords: ammonia volatilization, greenhouse gases, manure, sulfur

Abstract

A study was performed to assess: i) the feasibility to acidify the separated solid fraction of raw and co-digested pig slurry by using a powdery sulfur-based product and ii) the effect of this acidification method on greenhouse gases and ammonia emissions during manure storage. Samples of raw and co-digested pig slurry were collected at two commercial farms and mechanically separated by a lab-scale screw press device. The sulfur powder (80% concentration) was added to the obtained separated solid fractions at three application rates: 0.5%, 1% and 2% (w/w). Carbon dioxide (CO₂), methane (CH₄), nitrous oxide (N₂O) and NH₃ emissions were afterwards measured during storage of the acidified samples and compared to those measured from untreated samples (control). Gaseous emissions were determined with dynamic chamber method by Infrared Photoacoustic Detection. Gaseous losses were monitored along 30 and 60 days of storage time for raw solid fraction and digested solid fraction respectively. The addition of the tested sulfur powder to solid fractions showed to be a reliable and effective method to acidify raw and co-digested solid fractions. Results showed a significant reduction of both greenhouse gases and ammonia emission regardless of the separated solid fraction type. The highest sulfur application rate (2% w/w) led to a reduction of up to 78% of GHG emission and 65% of NH₃ losses from raw separated solid fraction when compared to control. Similar results were achieved from the co-digested solid fraction, with emission reduction of up to 67% for NH₃ and 61% for GHG.

Introduction

Gaseous losses of pollutants to the atmosphere are a major problem associated with animal manure management. In 2011 the agricultural sector contributed 94% to total ammonia (NH₃) emission in Europe (European Environment Agency, 2014). According to Oenema (2007), 52% of excreted N is available as crops nutrient, meaning nearly half excreted N is lost along the manure management chain. Barns and slurry stores represent up to 80% of the total NH₃ losses from agricultural activities (Anderson et al. 2003).

Greenhouse gas (GHG) losses from manure management, i.e. methane (CH₄), carbon dioxide (CO₂) and nitrous oxide (N₂O), account for 15% of total agricultural emissions in Europe (10% of total anthropogenic GHG emissions) (European Environment Agency, 2013). In recent years many studies validated methods for the reduction of GHG emission from manure storage such as floating covers (Balsari et al. 2013; Dinuccio et al. 2012; Balsari et al. 2006), natural crust (Sommer et al.
2000), perlite and lightweight expanded clay aggregate (Leca®) (Berg et al. 2006), wooden lids and chopped straw placed on the slurry surface (Amon, 2006).

A strategy widely used in Denmark to reduce NH₃ volatilization consists in slurry acidification (Eriksen et al. 2008). Ammonia volatilization can indeed be reduced by lowering slurry pH, whereby the NH₃/NH₄⁺ equilibrium shifts towards NH₄⁺ concentration. Acidification is also known to positively affect GHG emission (Fangueiro et al. 2014; Dai & Blanes-Vidal, 2013; Kai et al. 2008; Jensen, 2002; Frost et al. 1990; Stevens et al. 1989).

Nevertheless, acidification is commonly performed by using strong acids, mainly concentrated sulfuric acid. Some limitations to their use, such as their hazards to human health, are important issues that need to be overcome. Furthermore, at present, solutions to acidify solid manures (e.g. farmyard manure and slurry separated solid fraction) are lacking.

The paper presents the results of a laboratory study performed to assess: i) the feasibility to acidify the separated solid fraction of raw and co-digested pig slurry by using a powdery sulfur-based product and ii) the effect of this acidification method on greenhouse gases (CO₂, CH₄, N₂O) and NH₃ emissions during manure storage.

**Materials and methods**

*Manure sampling*

Samples of raw (RS) and co-digested slurry (DS) were sampled at two farms located in Piedmont (northwest Italy).

Raw slurry was collected from a pig-breeding farm, where 2500 sows and 2300 fattening pigs were bred on slatted floors. The pigs’ diet was mainly represented by corn mash, and to lesser degrees, of barley, soybean, wheat, and bran.

Co-digested slurry was collected in the same period from a CSTR (Continuous Stirred Tank Reactor) biogas plant with an installed electric power of 500 kW. The mesophilic plant (40°C) has a hydraulic retention time (HRT) of 40 days and is fed with (w/w) 70% pig slurry, 12% maize silage, 7% sorghum silage and 4% cattle manure.

Raw slurry and co-digestate were transported in 30 litres barrels to the laboratory and placed in a refrigerated room at +4°C until separation tests.

*Mechanical separation*

Separation tests were performed at the Waste Management Group laboratory of the Department of Agriculture, Forest and Food Sciences (DISAFA) - University of Turin, Italy. Raw slurry and co-digested slurry were mechanically separated by a lab-scale screw press device normally used to produce tomato sauce (Popovic et al. 2014). The machinery has a maximum working rate of 200 kg h⁻¹ of tomato, an auger rotation of 180 rpm and a 1 mm diameter mesh size. Screen openings of commercial mechanical separators vary, ranging from 0.1 to 3.0 mm, according to separator type and particularly to the total solids content of the input manure (Hjorth et al. 2010). For screw press, studies conducted in Italy by Balsari et al. (2006) and Dinuccio et al. (2014) reported screen...
openings ranging from 0.75 to 1.5 mm, with smaller openings typically used for pig slurry and larger for dairy cattle.

**Solid fraction acidification and chemical analysis**

The efficiency of acidification on emission abatement depends on the additive, manure type, step in the slurry management chain, and contact between additive and manure (Ndegwa et al. 2008). Several studies have confirmed that NH$_3$ emissions are directly related to the final pH of the slurry reached after the addition of the amendment (Fangueiro et al. 2014): by achieving pH values of 5.5 – 6 the NH$_3$ (Kai et al. 2008; Jensen, 2002; Frost et al. 1990; Stevens et al. 1989) and CH$_4$ (Ottosen et al. 2009) emissions can be reduced by 70 - 90%. Thus, we fixed the value 5.5 as pH target below which emission were expected to be decreased.

The obtained undigested (raw) and co-digested solid fractions were acidified by the addition of a powdery sulfur-based product (Microthiol® Disperss®, 80% micronized wettable elemental sulfur) widely used in crop protection as a fungicide. The rationale behind acidification with elemental sulfur relies on the chemical reaction described in equation (1):

$$S^0 + \frac{1}{2} O_2 + 2H_2O \leftrightarrow CH_2O + 2H^+ SO_4^{2-}$$  \hspace{1cm} (1)

It has been well documented (e.g., Fukumoto et al. 2003) that manure that is stored solid allows oxygen diffusion into the manure pile, and therefore, there will likely be the opportunity reaction of equation 1 to occur.

Sulfur was added to both co-digested and raw separated solid fractions at three rates: 0.5%, 1 % and 2% (w/w) calculated on wet basis (WB). Solid fraction after sulfur addition was thoroughly mixed manually to evenly distribute the powdery product. Unacidified raw and co-digested solid fractions were used as control. Prior to and after acidification, samples of all treatments were collected, and stored at 4°C prior to chemical characterization. Dry matter (DM) content was measured after drying (24h at 105 °C) the fresh samples to constant weight. The volatile solids content (VS) was calculated as weight loss upon ignition at 550 °C for 5h (VDI 4630, 2006). Samples were weighted using a four digits trusted balance (Kern®, mod. ABS 220-4). pH of the solid fractions prior and during the experiment were measured by a glass electrode for semi solid-biomasses (Hanna instruments® electrode HI 1053B). At the end of the trials pH was measured after solid fraction dilution in deionized water, followed by 45 min of shaking, and then 15 min of settling (Jorgensen & Jensen, 2009). Total N ($N_{tot}$) and total ammonia nitrogen (N-NH$_3$) were measured according to the Kjeldahl standard method (AOAC, 1990).

**Measurement of gaseous emission**

Emission tests were performed by filling 2000cm$^3$ jars with 1000cm$^3$ of unacidified and acidified solid fractions with three replicates per treatment. The bulk density of the tested raw and co-digested solid fraction samples was estimated to be 500g/1000cm$^3$. Gaseous (CO$_2$, CH$_4$, N$_2$O and NH$_3$) emissions were measured by a ventilated chamber system and using an infrared photo acoustic detector (IPD) (1412 Multi-gas Monitor, Innova® Air Tech Instruments, Ballerup, Denmark) as described by Dinuccio et al. (2008). The IPD was calibrated before the beginning of
the experiment by the manufacturer and was run with corrections from cross interferences between
CO$_2$-water vapor and measured target gases, and cross compensation (Huszár et al. 2008; Tirol-
Padre et al. 2014). Before emission measurements, each jar was closed with an airtight lid provided
with two ports for air inlet and outlet. The air inlet port was connected in an airtight way with a
flow meter and a pump. The headspace between the solid-fraction surface and the lid was then
ventilated to guarantee a complete air change per minute. Gaseous emission were monitored every
24h for the first 2 weeks of trial and three times per week thereafter. The operative steps followed
for emission measurement was carried out according to Dinuccio et al. (2008). Specifically, an air
flow of 1000 cm$^3$/min across the headspace was established for at least 20 min before gas sampling
to reach a steady state and then emissions were measured over a period of 16 min. Trials were
stopped when all (GHG and NH$_3$) gaseous emission dropped to zero for three consecutive days.
Specifically, the tests lasted 30 and 60 days for raw and co-digested solid fraction respectively. The
cumulative net gaseous emissions were determined according to Dinuccio et al. (2008). Data were
tested by one-way ANOVA and the Tukey tests ($\alpha = 0.05$). Measured gaseous losses were
converted into CO$_2$eq by using the IPPC (2013) Global Warming Potential (GWP) values. Along
the experiment, the environmental temperature at the laboratory was recorded by means of two
Onset® Hobo U12 data loggers.

### Results and discussion

#### Raw and co-digested solid fractions chemical characteristics

Chemical characteristics of raw and co-digested solid fraction samples measured at the beginning of
the experiments are shown in Table 1. Despite a similar DM content, VS concentration in the co-
digested solid fraction was significantly (p<0.05) lower than that in raw solid fraction, as a
consequence of organic matter degradation during the anaerobic digestion process. The initial pH
were 8.00 and 8.51 for the raw and co-digested solid fraction respectively. Also total nitrogen
concentration was similar for the two biomasses, whereas a higher ammonia nitrogen content was
found for the co-digested solid fraction, due to N mineralization occurring during anaerobic
digestion.

At the end of the experiment, the DM content of both raw and co-digested solid fraction were found
to be higher compared to the initial values (Table 2) as a consequence of water evaporation during
the experiments (Table 3). The evolution of solid fractions pHs along the experiments are shown in
Fig. 1. With respect to unacidified solid fraction from mechanical separation of raw slurry, pH
remained above 6.5 for the whole experimental period. Acidified fractions showed decreasing pH
values already after 24hrs from sulfur addition, as a result of H$^+$ formation and S oxidation (Roig et
al. 2004). Sample RS 2 reached the pH target (5.5) at day 7, RS 1 at day 9 and RS 0.5 after 14 days
from S addition. The minimum pH values (2.47) was reached by RS2 at day 23. With co-digested
solid fractions, sulfur addition took longer to affect solid fraction pHs. The latter started indeed to
drop after three days from acidification. This might be due to the higher buffer capacity of the
material when compared to non-digested solid fraction. DS 1 and DS 2 needed about a week to
reach the pH target, whereas pH of DS 0.5 treatment dropped below 5.5 after approximately 30
days. The minimum pH value (2.87) was reached by DS2 treatment at day 56.
Environmental temperature is well known to strongly affect NH₃ and CH₄ emissions (Dinuccio et al. 2008, Wang et al. 2014). Furthermore, Jaggi et al. (1999) found a strong influence of temperature on the rate of S oxidations. Recorded temperatures were similar along the two experiments. Average, minimum and maximum values are reported in Table 3.

**NH₃ emissions**

NH₃ emissions from raw-slurry separated solid fraction started to drop already at day 3 when pH approached 5.5 (Fig. 2a). All treatments were effective, regardless of S application rate. Fig. 3 shows the total NH₃ emission quantified along the trial. The control (RS) lost as ammonia approximately 6% of its initial total nitrogen content (Table 4). This value is consistent with that (5.6%) measured by Dinuccio et al. (2008). Acidified samples lost 1.93% to 3.20% of their initial total nitrogen (Table 4). Depending on the amount of added S, NH₃ emissions from RS 0.5, RS 1 and RS 2 acidified fractions were respectively 49%, 65% and 70% lower than those of the control treatment. Specifically, the higher the S application rate, the higher the abatement efficacy. These findings are consistent with measurement performed on raw slurry by Pain et al. (1990), Kai et al. (2008) and Dai & Blanes-Vidal (2013) by using sulfuric acid as acidifying agent. The NH₃ emissions from co-digested solid fraction were twofold higher when compared to those of raw separated solid fraction. This was probably due the higher initial N-NH₃ concentration (Table 1) (Chadwick et al. 2011). All acidification treatments significantly reduced ammonia emissions by approximately 65% with respect to untreated solid fraction. NH₃ emissions from acidified fractions started to drop at day 7 (Fig. 2b), when pH approached 5.5 (Fig. 1b). The control (DS) lost on average 19% of total initial nitrogen as ammonia, whereas from acidified solid fractions 6-7% of the initial nitrogen was lost as ammonia (Table 4).

**GHG emissions**

The effect of S addition on GHG emissions from the storage of raw slurry and co-digested solid fractions is displayed in Fig. 4. Although the two tests had different duration (30 and 60 days for RSs and DSs respectively) total GHG emission from control samples (RS, DS) were in the range of 6 kg CO₂eq m⁻². N₂O was the most relevant GHG in both experiments. Acidification showed to be more effective when applied on raw (non-digested) solid fractions. The highest emission reduction (%) was obtained with 2% S addition, but this value was found to be not significantly (p> 0.05) different from that (%) recorded for RS 1. A 0.5% S application rate was able to reduce by 44% GHG emission when compared to control. The same S application rate (treatment DS 0.5) increased CO₂eq emission by 44% when applied to co-digested solid fraction, due to high (+55%) N₂O emissions. Nevertheless, DS 0.5 reached the target pH very late (after 30 days from S addition), thus suggesting this rate to be too low for this kind of animal waste, being characterized by a high initial pH and buffer capacity. N₂O emissions increasing were probably due to an enhanced microbial activity with S as a growing factor (Sierra-Alvarez et al. 2007). Biogas obtained by digesting pig slurry generally contains approximately 3000-8000 ppm hydrogen sulfide (H₂S) (Wei-Chin Lin et al. 2012). Hydrogen sulfide have damaging effects on the engine components and equipment and it is therefore removed before combustion in combined heat and power units. The most common method for H₂S removal from biogas is based on the addition of a small amount of oxygen or air (3-5% v/v) directly into the digester (Ramos & Fdz-Polanco, 2014). In this way it takes place the biological aerobic oxidation of H₂S to elemental sulfur and sulphates by a
consortium of sulphur-oxidising microorganisms (e.g., Thiobacillus bacteria). This process can result in the accumulation of elemental sulfur and sulphates in the digester. It is assumed that in the co-digested slurry are present nitrifying prokaryotes that could have an affinity with sulfur. Therefore a higher sulfur dose might be necessary to inhibit the nitrification/denitrification biological activity of these bacteria that could be otherwise promoted by a lower S application rate.

Higher S application rates (DS 1 and DS 2) significantly reduced CO$_{2}$eq. losses (by 39% and 55% respectively). With special regards to methane, by applying 2% sulfur a 54% losses reduction was observed.

**Conclusions**

The addition of elemental S to solid fractions showed to be a reliable and effective method to acidify raw and digested solid fractions. Thus, it can be considered as an alternative method to the common sulfuric acid utilization. Sulfur addition led to significant reduction of gaseous losses (NH$_3$ and GHG) during the solid fractions storage. The most evident outcome is represented by the significant reduction of NH$_3$ emissions rate for both the tested biomasses with abatement of up to 70% in raw slurry solid fraction and 65% for the digested one.

GHG emissions were respectively reduced from 44% to 78% according to the amount of S added to non-digested solid fraction. The lower S rate significantly increased GHG emission from the digested solid fraction only with special regards to increased N$_2$O losses. The experimental results allow a first positive evaluation on the possibility to decrease the pH and gaseous emissions by adding sulfur to solid manures, thus enabling an effective pollution reduction without using strong acids. The latter aspect is indeed one of the main concerns and a major limit to the diffusion of manure acidification at a European level.

According to our preliminary results 1% S might be considered as the best application rate, allowing an emission reduction in line with the present acidification technology performances (Fangueiro et al. 2014). However, according to the current market price of powdery sulfur for crop protection purpose (1€/kg) the former application rate would cost around 5€ per ton of treated solid fraction. This cost is five times higher than that of slurry acidification by H$_2$SO$_4$. Nevertheless, it must be considered that besides the commercial powdery sulfur (normally used for crop protection), sulfur is a byproduct of the oil refining process. The latter is considered as a waste and thus its reuse in the animal waste management sector might considerably reduce the cost of slurry acidification. Appropriate procedures for safely using a powdery acidifying product are already under study by our research group. Moreover, the DISAFA-Waste Management Group is currently investigating the feasibility to lower solid fraction pH by S addition to slurry prior to mechanical separation, with the aim to i) reduce the S application rates and ii) acidify both solid and liquid fractions with a single treatment.

**References**


### Table captions

1. Average chemical characteristics of raw and co-digested solid fractions at the beginning of the experiments (n=3). Values of Standard deviation in brackets.

2. Average chemical characteristics of the solid fraction at the end of the test (n=3). Values of Standard deviation in brackets.

3. Average, maximum and minimum temperatures measured during the tests.

4. Percentage of ammonia nitrogen emitted from raw slurry SF and digested solid fractions, values with same letters are not significantly different, (n=3). Values of Standard deviation in brackets.

### Table 1.

<table>
<thead>
<tr>
<th>Biomass</th>
<th>DM (%)</th>
<th>VS (% DM)</th>
<th>pH</th>
<th>N&lt;sub&gt;tot&lt;/sub&gt; (%w/w)</th>
<th>N-NH&lt;sub&gt;3&lt;/sub&gt; (%N&lt;sub&gt;tot&lt;/sub&gt;)</th>
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<tr>
<td>Pig slurry</td>
<td>15.4 (0.16)</td>
<td>88.0 (0.14)</td>
<td>8.00 (0.01)</td>
<td>0.32 (0.09)</td>
<td>27.0 (0.07)</td>
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<tr>
<td>Co-digested</td>
<td>14.2 (0.32)</td>
<td>83.7 (0.33)</td>
<td>8.51 (0.02)</td>
<td>0.30 (0.10)</td>
<td>36.0 (0.11)</td>
</tr>
</tbody>
</table>

| DM: dry matter, VS: volatile solids, N<sub>tot</sub>: total nitrogen, N-NH<sub>3</sub>: ammonia nitrogen |

### Table 2.

<table>
<thead>
<tr>
<th>Sample</th>
<th>DM (%)</th>
<th>VS (% DM)</th>
<th>pH</th>
<th>N&lt;sub&gt;tot&lt;/sub&gt; (%w/w)</th>
<th>N-NH&lt;sub&gt;3&lt;/sub&gt; (%N&lt;sub&gt;tot&lt;/sub&gt;)</th>
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<tr>
<td>RS</td>
<td>23.4 (1.20)</td>
<td>85.2 (0.42)</td>
<td>7.14 (0.02)</td>
<td>0.54 (0.12)</td>
<td>1.00 (0.05)</td>
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<td>RS 0.5</td>
<td>27.5 (2.70)</td>
<td>84.9 (0.11)</td>
<td>6.18 (0.10)</td>
<td>0.77 (0.06)</td>
<td>45.0 (0.12)</td>
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<td>RS 1</td>
<td>28.5 (2.15)</td>
<td>80.6 (0.84)</td>
<td>4.65 (0.35)</td>
<td>0.88 (0.11)</td>
<td>53.0 (0.08)</td>
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<tr>
<td>RS 2</td>
<td>29.7 (2.51)</td>
<td>88.9 (0.51)</td>
<td>3.55 (0.28)</td>
<td>1.32 (0.08)</td>
<td>57.0 (0.12)</td>
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<td>DS</td>
<td>26.5 (1.76)</td>
<td>80.0 (0.44)</td>
<td>7.77 (0.11)</td>
<td>0.54 (0.05)</td>
<td>2.00 (0.00)</td>
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<tr>
<td>DS 0.5</td>
<td>30.1 (3.65)</td>
<td>80.1 (0.66)</td>
<td>5.88 (0.18)</td>
<td>0.68 (0.10)</td>
<td>30.0 (0.06)</td>
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<tr>
<td>DS 1</td>
<td>33.8 (3.64)</td>
<td>80.7 (0.49)</td>
<td>4.63 (0.36)</td>
<td>0.85 (0.14)</td>
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<td>DS 2</td>
<td>36.1 (2.19)</td>
<td>81.1 (0.44)</td>
<td>3.51 (0.23)</td>
<td>0.92 (0.10)</td>
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### Table 3.

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<th>Max (°C)</th>
<th>Min (°C)</th>
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<td>24.1</td>
<td>15.5</td>
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<tr>
<td>Co-digested slurry</td>
<td>21.3</td>
<td>26.5</td>
<td>15.5</td>
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<tr>
<td>Trial</td>
<td>N_{NH_3} emitted (%N_{tot})</td>
<td></td>
<td></td>
</tr>
<tr>
<td>---------</td>
<td>-----------------------------</td>
<td></td>
<td></td>
</tr>
<tr>
<td>RS</td>
<td>6.34 (0.94) b</td>
<td></td>
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<tr>
<td>RS 0.5</td>
<td>3.20 (0.20) a</td>
<td></td>
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<tr>
<td>RS 1</td>
<td>2.24 (0.05) a</td>
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<td>RS 2</td>
<td>1.93 (0.40) a</td>
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<tr>
<td>DS</td>
<td>19.2 (1.08) b</td>
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<tr>
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<td>7.27 (0.84) a</td>
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<td>DS 1</td>
<td>6.09 (0.55) a</td>
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<tr>
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**Figures captions**

Fig. 1. Average pH values pattern of (a) raw slurry solid fraction and (b) co-digested solid fraction samples along the experiment.

Fig. 2. Emission fluxes of ammonia (NH$_3$) during storage of (a) raw slurry solid fraction and (b) co-digested solid fraction. Error bars indicate standard deviation (number of observations=3).

Fig. 3. Total net ammonia emission from (a) raw slurry solid fraction and (b) digested solid fraction (values with same letters are not significantly different).

Fig. 4. Total GHG emissions during a) raw pig slurry solid fractions and b) co-digested solid fractions storage (values with the same letters are not significantly different, n=3).
**Fig. 2.**

**Fig. 3.**

**Fig. 4.**