



Country report

Impact of slurry removal frequency on CH₄ emission and subsequent biogas production; a one-year case study

Lu Feng^{a,b,*}, Lise Bonne Guldborg^b, Michael Jørgen Hansen^b, Chun Ma^c, Rikke Vinther Ohrt^b, Henrik Bjarne Møller^b

^a Division of Environment and Natural Resources, Norwegian Institute of Bioeconomy Research (NIBIO), 1431 Ås, Norway

^b Department of Biological and Chemical Engineering, Aarhus University, Blichers Allé 20, 8830 Tjele, Denmark

^c Department of Agroecology, Aarhus University, Blichers Allé 20, 8830 Tjele, Denmark



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ABSTRACT

Anaerobic digestion of animal slurry to produce biogas is the dominated treatment approach and a storage period is normally applied prior to digestion. Pre-storage, however, contributes to CH₄ emissions and results in loss of biogas potential. Manure management was found to be an efficient approach to reduce not only the on-site CH₄ emission but may also have extended influence on CH₄ emission/losses for storage and subsequent biogas process, while the connection remains unclear. The objective of this study was therefore to evaluate the impact of slurry management (e.g. removal frequency) on CH₄ emission (both on-site and storage process prior to biogas) and biogas yield. An experimental pig house for growing-finishing pigs (30–110 kg) and the relevant CH₄ emission was monitored for one year. In addition, the specific CH₄ activity (SMA) test was conducted and used as an alternative indicator to reflect the impact. Results showed that the manure management affected both on-site and subsequent methane emission; with increased manure removal frequencies, the methane emission became less dependent on variation of temperatures and the specific methanogenesis activity was significantly lower. The highest SMA (100 mL CH₄ gVS⁻¹), for instance, was observed from the slurries with limited emptied times, which was 10 times of that from the slurries being emptied three times a week. These findings could enlighten the development of environmentally friendly strategies for animal slurry management and biogas production.

1. Introduction

Intensified livestock production is an important source of GHG emissions, while CH₄ has much higher global warming potential (28 times more powerful than CO₂) (Gerber et al., 2013). Manure management was estimated to be about 10% of the total CH₄ emissions (in total, the CH₄ emission from the agricultural sector was 3.5Gt CO₂eq in 2018) from agriculture and led to increased awareness of the releasing of CH₄ from animal waste production facilities (Hill et al., 2001; Serrano-Silva et al., 2014). Therefore, it is important to develop an effective approach to mitigate the CH₄ emission and diminish its negative influence.

CH₄ emission from manure storage is affected by various issues, for instance, storage temperature, feeding composition, and ventilation (Elsgaard et al., 2016). Among these, the manure removal strategy and the storage duration play important role (Philippe and Nicks, 2015); long-term storage could enrich the methanogenic community and thus elevate the overall CH₄ emission (Møller et al., 2004), especially with a

portion of remaining manure due to incomplete removal acting as ‘inoculum’ (Ngwabie et al., 2016). Frequent emptying of slurry pits could wash out of active methanogens, which is considered as a good method to reduce the CH₄ emission (Dalby et al., 2021). Guarino et al. (2003) reported a reduction of 19% in total CH₄ emission when the manure in a pig house is removed weekly compared to a traditional deep-pit system. Lavoie and IRSST (2006) observed 14% reduction on CH₄ emission when manure was removed three times a week instead of only once.

Currently, anaerobic digestion (AD) is one of the most dominant approaches for treatment of animal slurry. In Denmark, for instance, the Ministry of Climate and Energy proposed a ‘Green Growth’ initiative aiming at using 50% of animal manure for biogas production, representing an increase of biogas production of approx. 15 PJ/year to a total of approx. 20 PJ/year in 2020 (Bundgaard et al., 2014). As a green process, it is of great importance to minimize the methane emission from the processes relevant to biogas activities, which could also add revenue

* Corresponding author at: Division of Environment and Natural Resources, Norwegian Institute of Bioeconomy Research (NIBIO), 1431 Ås, Norway.
E-mail address: lu.feng@nibio.no (L. Feng).

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Table 1
Characteristics of pig slurries during the entire investigating period.

Parameters ¹	PS-0 (Reference, 40 days) ⁴				PS-1 (7 days) ⁴				PS-2 (2 days) ⁴			
	1	2	3	4	1	2	3	4	1	2	3	4
TS (%)	5.99	7.72	7.66	8.73	9.09	8.79	9.09	8.04	9.44	8.43	9.09	8.97
VS (%)	4.55	5.99	6.01	7.007	6.93	6.88	6.71	6.47	7.56	6.35	7.25	7.25
pH	6.98	6.90	6.60	6.66	6.93	6.88	6.71	6.58	6.71	6.76	6.67	6.64
VFAs (mg/L)	1.4 × 10 ⁴	1.3 × 10 ⁴	1.4 × 10 ⁴	1.3 × 10 ⁴	1.2 × 10 ⁴	1.1 × 10 ⁴	1.1 × 10 ⁴	1.3 × 10 ⁴	1.4 × 10 ⁴	1.4 × 10 ⁴	1.3 × 10 ⁴	1.4 × 10 ⁴
NH ₄ -N ⁺ (g/L)	2.65	2.72	4.33	3.48	2.77	2.98	2.9	2.67	3.41	3.18	3.29	2.89
TKN (g/L)	3.49	3.99	5.63	5.33	4.99	4.75	4.7	4.80	5.57	4.96	5.23	5.26
SMA40d (ml CH ₄ gVS ⁻¹)	9.09 ± 0.23				10.18 ± 0.17				8.45 ± 0.27			
SMA40d mesophilic (ml CH ₄ gVS ⁻¹)	104.64 ± 0.02				33.55 ± 4.25				10.99 ± 0.48			
CH ₄ emission (kg CO ₂ eq./ton pig slurry)	4.67	7.78	5.14	2.94	4.58	5.94	4.56	4.30	4.92	7.16	4.42	6.03
CH ₄ emission (Nml CH ₄ gVS ⁻¹)	2.22	2.81 ± 1.73	1.85 ± 0.93	0.91 ± 0.43	1.43 ± 0.28	1.87 ± 0.45	1.47 ± 0.82	1.44 ± 0.34	1.41 ± 0.43	2.44 ± 0.36	1.32 ± 0.37	1.80 ± 0.41
CH ₄ /(CH ₄ + CO ₂) (%)	34.21 ± 16.0	40.15 ± 6.85	24.36 ± 10.07	20.79 ± 4.04	26.84 ± 1.99	31.61 ± 4.35	22.46 ± 4.01	24.25 ± 3.62	27.39 ± 1.80	28.68 ± 2.37	21.77 ± 2.89	23.02 ± 1.83
Average Temperature (°C)	17.3 ± 1.8	21.7 ± 1.4	20.0 ± 0.9	17.4 ± 0.4	–	–	–	–	–	–	–	–

1. Average value of entire period; 2. Test Period: 1: 2020. Jun-Aug; 2: 2020. Sep-Nov; 3: 2020.Nov-2021.Jan; 4: 2021. March-May; 3 The storage temperature was measured from reference unit, while the difference between sections was measured manually which was negligible; 4. Removing frequency: PS-0, 40 days; PS-1, 7 days; PS-2, 2–3 days.

for biogas plants and make them more sustainable (Im et al., 2020). Normally, the animal slurry is stored for 10 to 150 days in storage tanks before its utilization (for biogas), leading to CH₄ emission and losses of biogas production (Shin et al., 2019). According to Feng et al. (2018), CH₄ losses accounted for 1–2% of total CH₄ potential during the slurry storage prior to biogas. However, the impact of in-house slurry management on its following CH₄ emission/subsequent biogas production remains unclear.

The aim of the presented study was to investigate the influence of slurry management, e.g. slurry removal frequency, on CH₄ emission and biogas potential. Specific focus was aimed at the CH₄ emission generated between the pig house and biogas process, including on-site emission, and subsequent processes of transportation and storage. The CH₄ emission rate from the pig slurry was monitored following four fattening periods in a year (June, 2020 to May, 2021), and methanogenic activities tests were also implemented.

2. Materials and methods

2.1. Experimental set-up

Three pig houses at Aarhus University, Foulum, Denmark, were used in this study. During the experiment, the slurry in the experimental unit 1 (PS-1) and unit 2 (PS-2) was either removed weekly or emptied automatically three times a week (Monday, Wednesday and Friday) once the slurry reaching the specified level. Slurry from the reference unit (PS-0), however, was only emptied twice (first at day 40 and second in the end) during the entire fattening period. The experiment lasted for one year and covered four consecutive fattening periods. In each period, the pig slurry was collected four times on days 20, 40, 60, and 77. In PS-0 slurry samples (ca. 10 L) were collected as a split volume during emptying of the slurry channels at day 40 and 77 and manually from the slurry channel at day 20 and 60. In PS-1 slurry samples (ca. 10 L) were collected as a split volume during emptying of the slurry channels. In PS-2 samples were collected as a split volume (c.a. 10 L) during circulation of the slurry in the slurry funnels. The slurry temperature was measured and recorded during sampling. The total volume of slurry emptied each time was recorded. The slurry was transported shortly (less than one hour) to the laboratory to monitor the CH₄ emission and gas

composition. The testing temperature was controlled in accordance with the slurry temperature measured from the pig house, while the remains of each slurry sample was stored at –18 °C prior to analysis of VFAs, ammonia, pH, etc.

2.2. Measurement of CH₄ emission rate and activities (SMA)

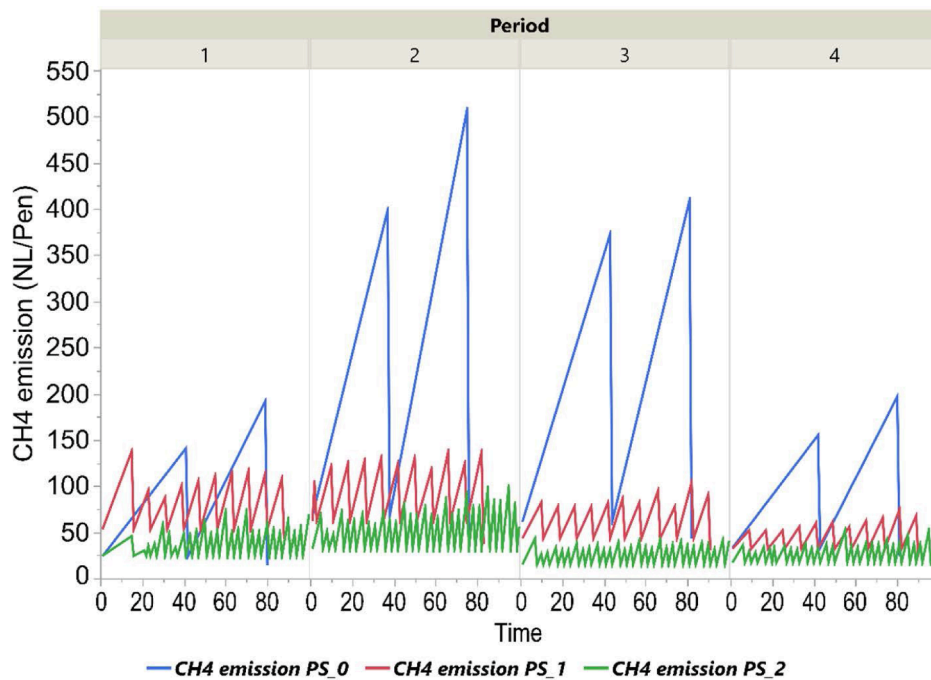
CH₄ emission test was set up using 500 mL infusion bottles. Three infusion bottles were prepared as replicates for each tested pig slurry. 400 g of pig slurry were added to each bottle, which were then tightly sealed with rubber stoppers and screw caps. All bottles were flushed with N₂ for two minutes to replace the headspace air and incubated in a water bath to control the temperature. The CH₄ emission rate was continuously monitored using AMPTS II (Bioprocess®, Sweden).

SMA test were performed to measure the activity of indigenous methanogens following the protocols suggested by Shin et al. (2019). Certain amounts of pig slurry was added into glass serum bottles (working volume 500 mL) by mixing with deionized water to adjust the VS concentration to around 2 gVS/L. 2.0 g COD/L of sodium acetate was used as a substrate, and NH₄Cl, KH₂PO₄, and FeCl₂·4H₂O were added to yield a COD:N:P:Fe ratio of 100:5:1:0.33 (Kim et al., 2006). In addition, trace nutrients were added like the followings (in mg/L): NaHCO₃ 1000; MgCl₂·6H₂O 100; CaCl₂·2H₂O 75; Na₂MoO₄·4H₂O 0.01; H₃BO₃ 0.05; MnCl₂·4H₂O 0.5; ZnCl₂ 0.05; CuCl₂ 0.03; NiCl₂·6H₂O 0.05; CoCl₂·2H₂O 0.5; Na₂SeO₃ 0.05. The bottles were sealed with rubber stoppers, secured with aluminum crimps and placed in an incubator after flushing the headspace of bottles with N₂ gas. The SMA test lasted for 40 days, as suggested by Kim et al. (2006).

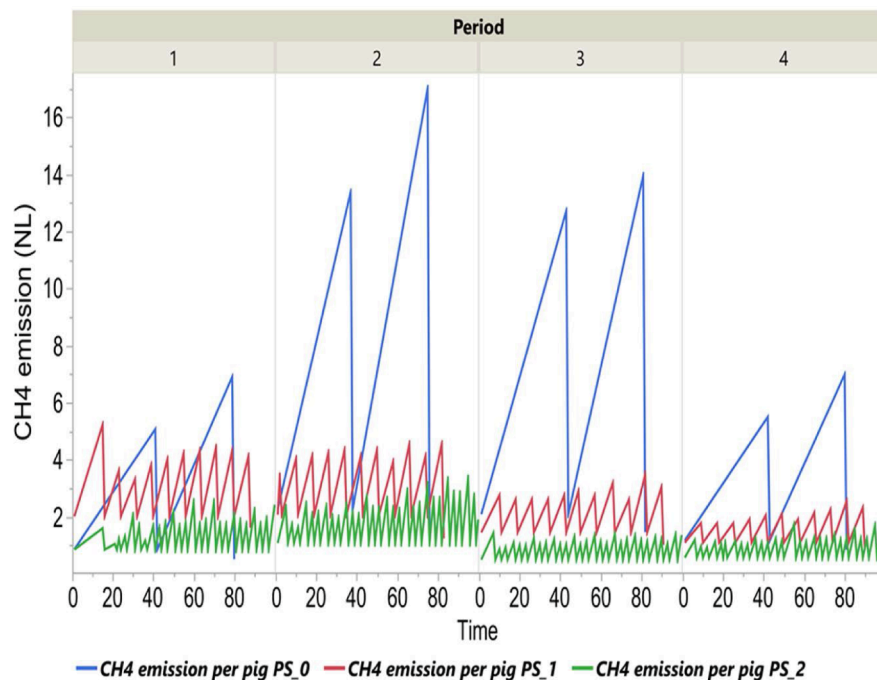
2.3. Modelling of in-house CH₄ emission

The measured CH₄ emission rate was used for modelling of the CH₄ emission in the pig house. Under the assumption that the increase of the pig slurry volume increases linearly, the volume of pig slurry (on any given date) was calculated based on the data from each emptying (Eq. (1)).

$$y = b + \frac{\Delta y}{x} \quad (1)$$



(a). CH₄ emission per pen



(b). CH₄ emission per pig

Fig. 1. Prediction of in-house CH₄ emission corresponding to removal frequencies (a. CH₄ emission per pen; b. CH₄ emission per pig).

y represents the current slurry volume (m^3) at a given date, b is the volume of pig slurry before the given date. Δy represents the difference (volume, m^3) between the most recent two emptying of slurries (as the emptied volume varied each time). x .

The equivalent weights (m) of the slurry volumes were estimated according to Eq. (2):

$$m = y \cdot \rho \tag{2}$$

While y represents the current slurry volume and ρ is the density of

pig slurry (1.03 g/mL^3) (Kowalski et al., 2013).

Thus, the in-house CH₄ emission rate was estimated according to Eq.3:

$$CH_4 \text{ emission} = \frac{m \cdot VS}{100} \cdot CH_4 \text{ emission rate} \tag{3}$$

where CH₄ emission represents the total daily CH₄ emission (NmL), which is calculated based on the slurry VS (%) and measured CH₄ emission rate (NmLCH₄. gVS^{-1}). In addition, the CH₄ emission per pig is

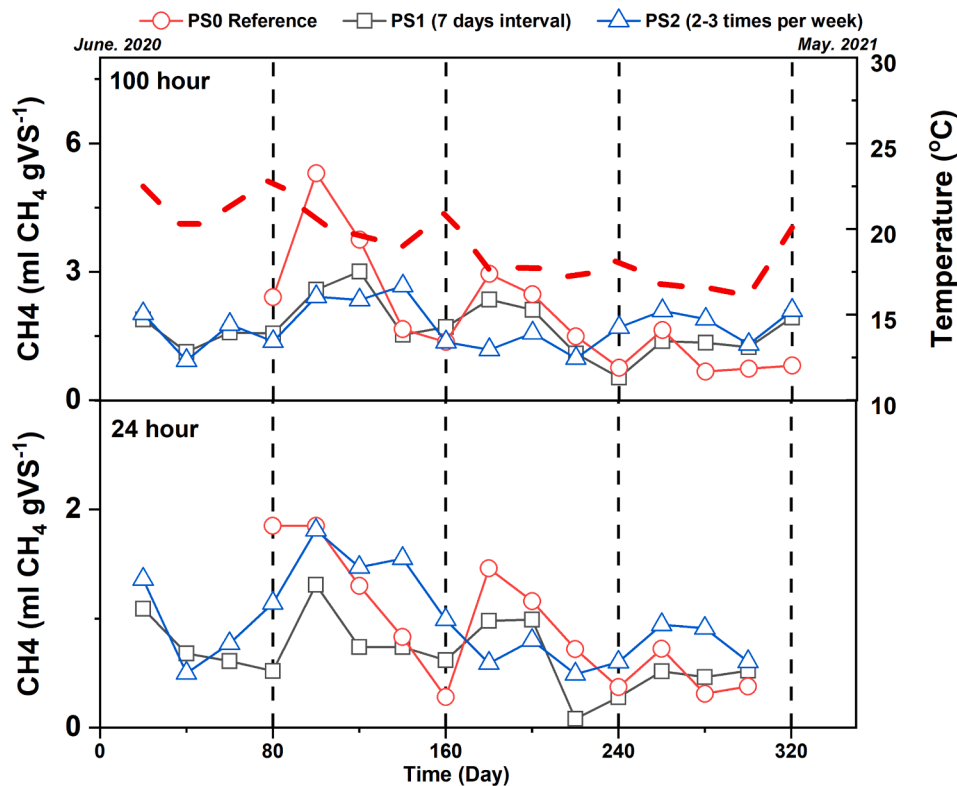


Fig. 2. Variation of (cumulative) CH₄ emission rate within 24 and 100 h (The first data point from day 40-PS-0 was neglected as it contained flushing water).

simply calculated by dividing the CH₄ emission acquired from Eq.3 by the (average) numbers of pigs per pen.

2.4. Analyses

2.4.1. Slurry analysis

TS and VS were measured according to standard methods (APHA, 2005). Dissolved VFA was determined using a gas chromatograph (Agilent technologies 7890A, CA 95051, USA), equipped with a flame ionisation detector (FID) and helium as the carrier gas. A DB-1 Column with a length of 30 m and inside diameter of 0.53 mm was used. The temperatures of initial oven, injector port, and detector were 100, 285, and 300 °C, respectively. The following temperature programming of oven was set: 100 °C hold 1 min, ramp to 120 °C at 10 °C min⁻¹, hold 5 min; ramp to 220 °C at 30 °C min⁻¹, hold 3 min. TAN was determined weekly from digestate using photometry (Spectroquant Kit, Merk, NJ, USA). Total Kjeldahl nitrogen (TKN) was determined according to APHA (2005). pH was measured using a Portamess 911 pH meter (Knick, Berlin, Germany).

2.4.2. Gas analysis

Gas composition was determined periodically using gas chromatography (Agilent technologies 7890A, CA 95051, USA) equipped with a thermal conductivity detector (TCD) and helium as the carrier gas. Alltech® CTR 1 double column (Grace, Maryland 21044, USA) was used. The temperature of oven, injector port, and detector was 120, 150, and 150 °C, respectively.

2.5. Data acquisition and graphing

OriginPro 2018 (OriginLab, MA 01060, USA) and JMP 14.0 (SAS Institute Inc, 10,740 Cary, USA) were used for graphing, data treatment, and statistics analysis. Gas production was automatically monitored and recorded using AMPTS II (Bioprocess, Sweden).

3. Results and discussions

3.1. Characteristics of pig slurries

Table 1 lists the main characteristics (TS, VS, pH etc.) of the pig slurries. In general, there is no significant difference observed between sections. Pig slurries had TS/VS within the range of 6.0–9.4% and 4.6–7.6%, with that of PS-0, 1, and 2 determined to be 7.5/5.9%, 8.8/6.7%, and 9.0/7.1%, respectively. pH was similar as well, with the average pH of 6.7 from all sections. It should also mention that the lower TS/VS from control section (PS-0) at the start was due to the leakage of flushing water.

3.2. In-house CH₄ emission

The predicted in-house CH₄ losses corresponding to various slurry removal frequencies is shown in Fig. 1. In general, the in-house CH₄ losses varied among sections as the total slurries level were not the same due to the different removal frequencies. In addition, the in-house loss varied among investigating periods with significantly higher CH₄ emission observed from period 2 due to the relatively high temperature (above 20 °C, on average) (Table 1). The patterns of accumulated CH₄ emission followed the level changes due to slurry generation and removing, which was accumulated linearly and peaked prior to the next empty. As shown in Fig. 1, it is clearly that increasing the slurry removal frequencies is an efficient approach to mitigate the in-house CH₄ emission. Within all investigating periods, the CH₄ emission from control section (PS-0) peaked between 150 and 500 NL CH₄ per pen, corresponding to 15 to 50 NL CH₄ per pig, while that from PS-1 and PS-2 peaked at around 100–150 NL and 25–100 NL CH₄ per pen (2–5 and 1–2 NL per pig, respectively). It should also be noted that the in-house emission was predicted based on the CH₄ emission rate measured indirectly. This explains why the CH₄ emission of PS-0 at the start of period 1 was lower than that from PS-1 at the start of period 1 (Fig. 1) due to the unexpected water leaking into the slurry stream that diluted the slurry.

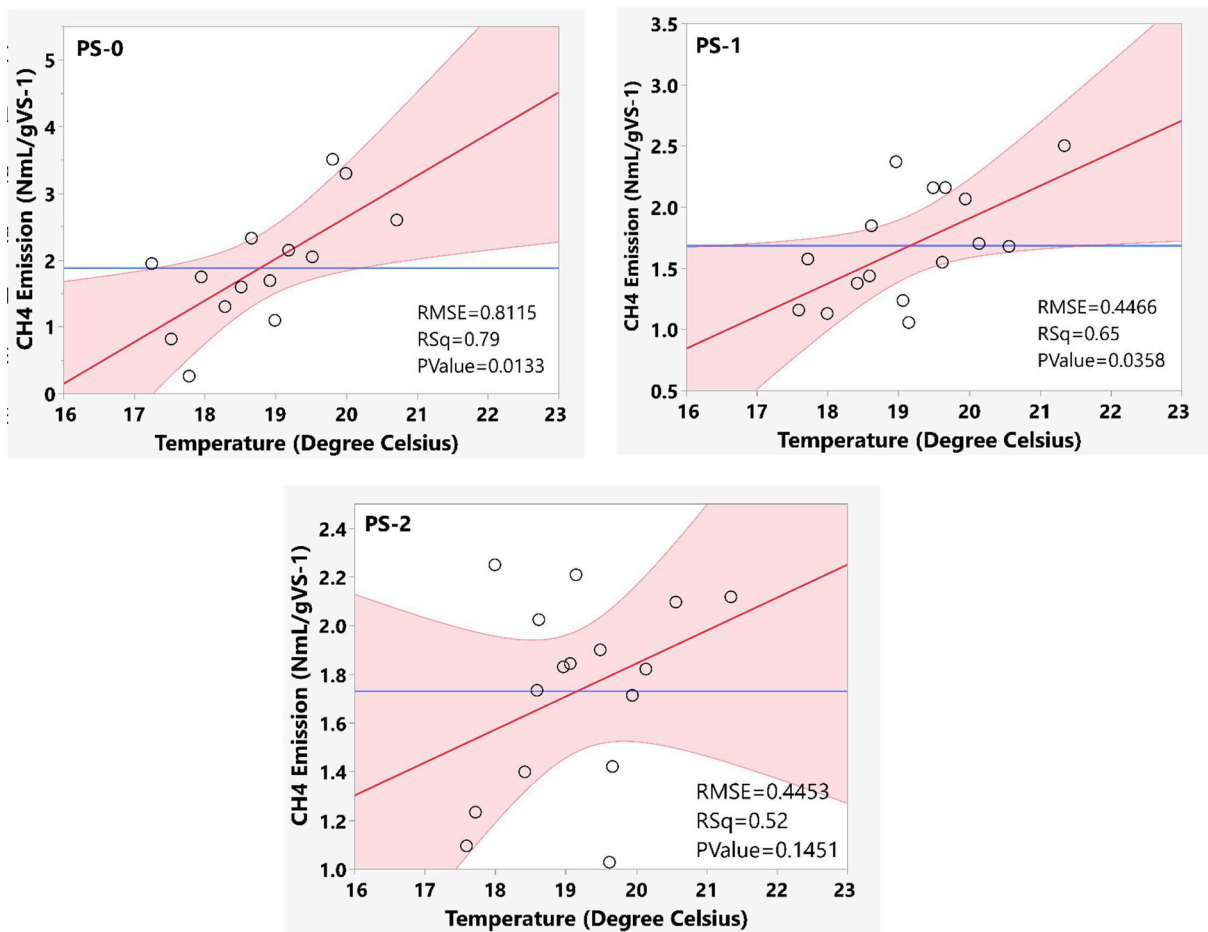


Fig. 3. Least squares regression analysis of CH₄ emission rate (100 h' data) and (in-house) storage temperature.

The unexpected input of water continued until the first emptying of PS-0 (Day 40).

3.3. Impact of removing frequencies on subsequent CH₄ emission rate

As shown in Fig. 2, the variations of CH₄ emission rate follows the change in slurry temperature and fattening periods, but is less affected due to the removal frequencies (either for short (24 h) or long term (100 h) observation). The 100 h as 'long-term' was decided according to the average storage time (4–5 days) in the full-scale biogas plant located in Aarhus University, Foulum (Tjele 8830, Denmark), while to monitor the CH₄ emission taking place within 24 h could give an idea on how fast it is emitted. For each investigating period, the CH₄ emission rate peaked at the start and declined over time, which is probably related to the feeding composition and growth of pigs. There are few exceptions observed but this was in general due to the increased slurry temperature. For instance, the cumulative CH₄ emission sampling at day 320 was higher than day 280, as the slurry temperature increased from 16 to 20 °C. According to the test, 50% of the CH₄ (considering 100 h as 100%) was emitted within the first 24 h.

It seems that the CH₄ emission rate is not significantly affected by the removal frequency. This is in agreement with Dalby et al. (2021) who reported that the microbial enrichment or 'inoculum' has only limited impact on CH₄ emission, while the storage temperature in general still plays a key factor on the activities of methanogenesis (Kariyapperuma et al., 2018). In this case study, the pig slurry was stored at temperature ranging between 17 and 22 °C, which are below the optimal temperature for methanogenesis (Feng et al., 2018). At low temperature, methanogenesis activities are rather lower, therefore becomes the rate-limiting

step, which could partly explain the similarity of CH₄ emission between sections. In practice, slurries with longer periods between removals still have higher in-house GHG emission due to the larger amount of slurry present (Fig. 1). Indeed, the BMP test showed no significant difference between slurries emptied with different time intervals (data not shown). In total, the CH₄ emission due to storage accounted for 1–2 % of total CH₄ potential (BMP). Therefore, the differences caused by the removal frequencies might be not detectable with the regular BMP test.

However, there might still be indirect impacts that could be investigated, for instance, the correlations between the CH₄ emission and temperature due to the removal frequencies. In order to achieve this, least squares regression analysis was implemented (Fig. 3). CH₄ emission rate from the control section fits best with the storage temperatures, while that from section 2 is the least relevant ($R_{PS-0}^2 > R_{PS-1}^2 > R_{PS-2}^2$). Referring back to the hypothesis, the methanogenic activity might be enriched in the control section but was 'hidden' under lower storage temperature. Thus, SMAs tests were carried out and to use as an indirect indicator.

3.4. SMA test and VFAs profile

As shown in Fig. 4a, the measured SMAs are very similar between sections (almost 10 mL CH₄ gVS⁻¹) at low temperature (15 °C) but presents significant differences patterns at 30 °C, with the only exception observed from PS-2. The highest SMA was observed from PS-0 at 30 °C, which was more than 100 mL CH₄ gVS⁻¹, followed by PS-1 (c.a 35 mL CH₄ gVS⁻¹). PS-2, however, had similar SMAs (around 10 mL CH₄ gVS⁻¹) at all testing temperatures. VFA profiles before and after SMA

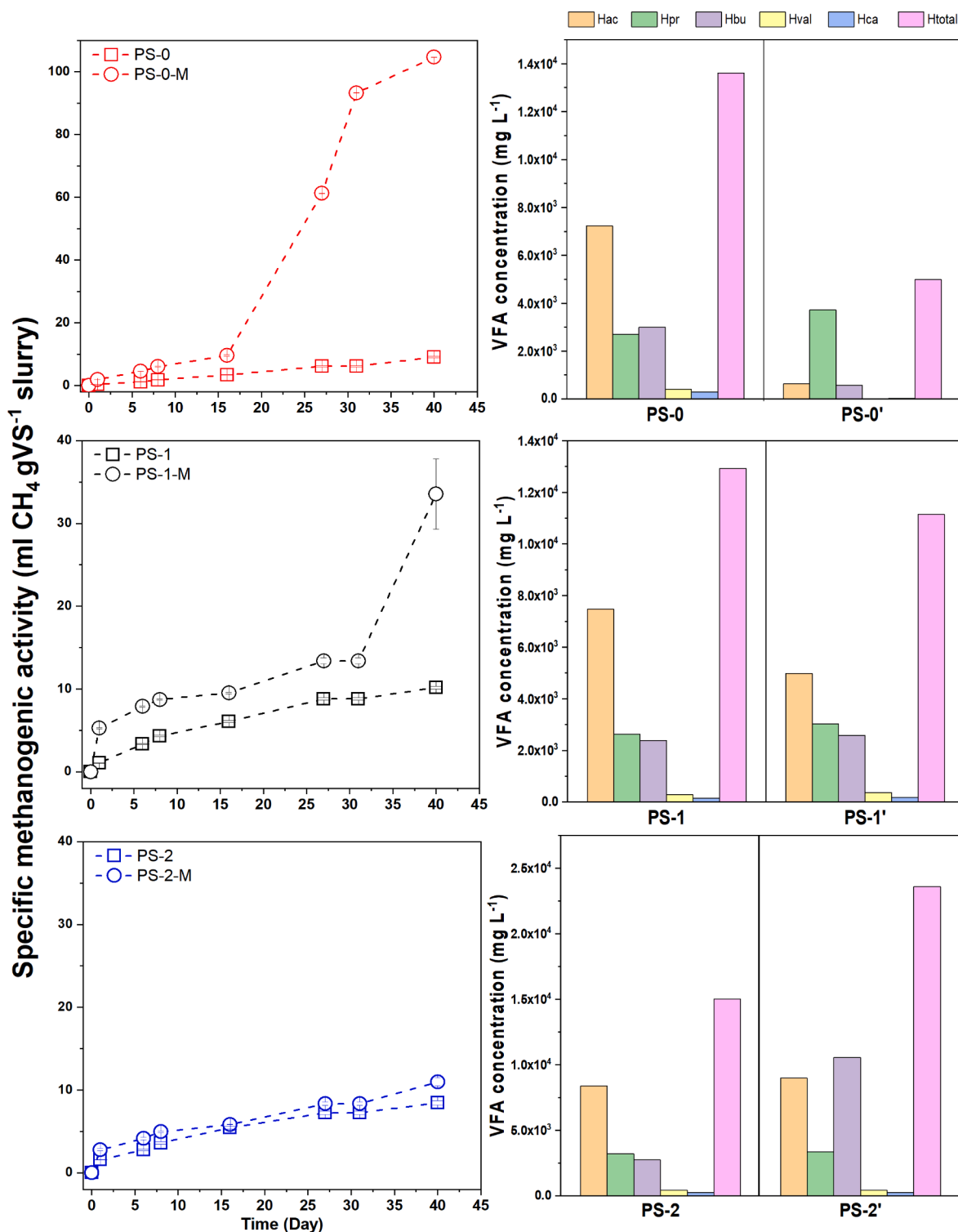


Fig. 4. SMA results (a) CH₄ emission rate and changes of VFAs profile and (b). CH₄ content measured from SMA test. (1. Removal frequencies = 2 (PS-2), 7 (PS-1), and 40 days (PS-0); 2. Only the control sector was measured under four temperatures (15, 20, 25 and 30 °C). 3. SMA was carried out using samples collected at the end of winter trials; 4. Hac, acetic acid; Hpr, propionic acid; Hbu, butyric acid; Hval, valeric acid; and Hca, caproic acid.).

tests were examined (Fig. 4, right), while there were noticeable differences among sections: VFAs from PS-0, for instance, experienced a large reduction in concentration from $1.4 \times 10^4 \text{ mg L}^{-1}$ to below $0.5 \times 10^4 \text{ mg L}$, accounting for over 63% of total VFAs, while that from PS-1 dropped slightly from 1.3 to $1.1 \times 10^4 \text{ mg L}^{-1}$, corresponding to 14% of reduction. However, the total VFAs of PS-2 increased from 1.5 to $2.4 \times 10^4 \text{ mg L}^{-1}$

(57% more) as a result of butyric acid accumulation (Fig. 4). Butyrate, with its isoform, isobutyrate, is normally used as an indicator of process stress and failure in anaerobic digestion (Ahring et al., 1995), indicating that the CH₄ emission is highly inhibited. SMA tests were repeated using the samples from the end of the experiment and tested at more temperatures (15, 20, 25, and 30 °C) for PS-0, with the aim of gaining more

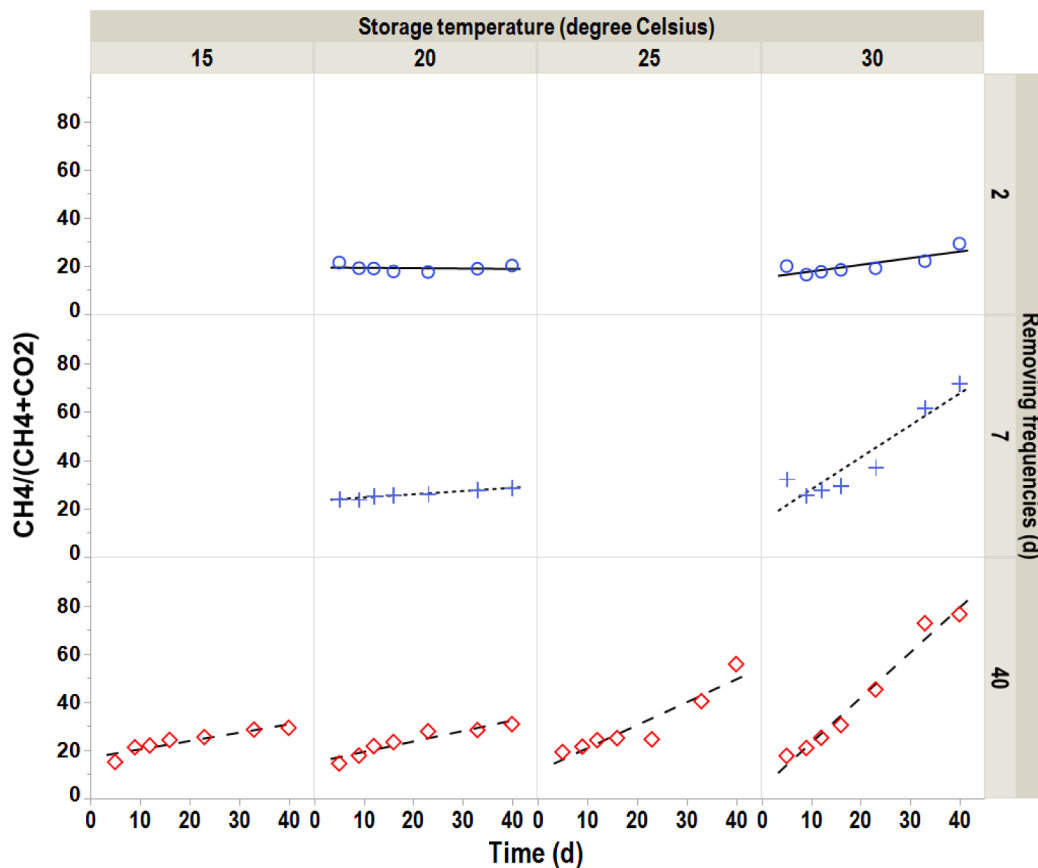


Fig. 4. (continued).

information regarding SMAs corresponding to temperature. In the second SMA test, the result was in accordance with the 1st test (Fig S2). CH₄ contents from PS-0 and PS-1 were in the range of 20–30% below 20 °C and started to rise dramatically at temperatures over 25 °C (Fig. 4b). PS-2, however, had similar CH₄ content (ca. 20%) at both temperatures. According to Liu and Whitman (2008), a short storage time (frequent removal) could be a potential strategy to reduce CH₄ emission, as methanogenesis requires accumulation of sufficient amounts of suitable substrates, such as acetate, H₂/CO₂, or formate. In this study, the animal slurry was stored at low temperature, therefore the ‘inoculum’ effect due to long-term storage is not as high as normal. Moreover, the CH₄ concentrations increased to a greater extent at higher temperature (30 °C) from PS-0 (which had higher methanogenic activities), leading to huge differences between sections.

4. Conclusion

The study evaluated the impact of removal frequencies on CH₄ emission from pig slurries prior to a biogas production process. Within a one-year period, the pig slurry tanks were emptied with different frequencies from two days to forty days, while the pen that was emptied least led to a higher methane level inside the pig house associated with the relatively high mass of accumulated slurry but had no significant impact on the CH₄ emission rate (per unit VS). However, the CH₄ activities (SMA) were significantly reduced with increased removal frequencies, indicating the CH₄ emission/loss became less dependent on the temperature and the potential CH₄ emission/losses at high temperature could be minimized. Therefore, it is an approach that could potentially reduced the methane losses for subsequent biogas production processes.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary material

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.wasman.2022.06.024>.

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