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Biomethane yield of novel source segregated dairy excreta under different anaerobic digestion systems

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A B S T R A C T

Source Segregation (SS) is a novel strategy in dairy housing that can reduce emissions and separate organic matter and nutrients more efficiently than traditional slurry solid-liquid separation. The anaerobic digestion (AD) methane yield of the SS fractions, however, is unknown. We aimed at unveiling the biomethane yield of these fractions by conducting AD experiments under different configurations: batch, continuous feeding, and fed-batch. In the batch test, the solid (SF) and liquid fraction (LF) from the SS system, a slurry collected from the pit (CS), and a self-made slurry (MF) were used as substrates. The results showed that the specific CH_4 yields of the SF and MF were in same range and both higher than the CS. We concluded that SS can increase the CH_4 yield of dairy excreta mainly by reducing losses in the animal house. The SF and MF were then compared in a continuously-fed thermophilic test, where SF had a higher specific (174 compared to 105 NL kg^{-1} VS) and volumetric (12.2 compared to 9.9 NL CH_4 kg^{-1} excreta) yields. We concluded that the SF can effectively substitute slurry in AD without compromising the yield, possibly increasing economic viability by reducing transport costs and reactor size. Further, SF produced 356 NL CH_4 kg^{-1} VS and a digestate with 1.8% lower dry matter in the fed-batch as compared to continuous feeding. Continuously stirred fed-batch can thus increase the CH_4 yield of the SF and reduce the DM of its digestate potentially contributing to lower emissions in storage and field application.

1. Introduction

Source segregation (SS) of faeces and urine is a novel manure collection strategy emerging as an alternative to slurry collection systems where faeces and urine are mixed in the underground pit of the animal house. Source segregation involves scraping faeces outside the animal house while collecting urine separately in the pit, which aims at reducing in-house greenhouse gas (GHG) and ammonia emissions, and separating nutrients to facilitate their recycling for crop fertilization (Swierstra et al., 2001; Aarnink et al.,

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2007; Vu et al., 2016; Galama et al., 2020). Few SS systems were described for dairy housing and were found to have 94%, 23% and 94% separation efficiencies of organic matter (OM), total ammoniacal nitrogen (TAN) and phosphorus (P), respectively, being higher than those allowed by traditional solid-liquid separation of slurry (EL Mahdi et al., submitted for publication). Further, SS was found to reduce NH_3 emissions in the animal house by up to 49%, respectively, as compared to slurry systems (Swierstra et al., 2001; Lachance et al., 2005). Hence, SS is proposed as a more environmental alternative to slurry collection systems. While SS systems are quite well described for pig housing, they are newly emerging in dairy housing and little is known about their implications on the different steps of the manure management chain including anaerobic digestion.

Anaerobic digestion (AD) is a common technique used in manure management. AD is a process converting organic material into biogas, a mixture of mainly methane and carbon dioxide, that can be used as a renewable energy source. AD of slurry also produces digestate as a liquid by-product, which is agronomically valuable because of its content of nutrients (e.g. nitrogen (N), phosphorus (P), potassium (K) and micronutrients) and stable carbon (Risberg et al. 2017; Barłóg et al., 2020). In addition, anaerobically digesting slurry is known to reduce GHG from slurry storage and soil application (Clemens et al., 2006; Marañón et al., 2011; Yao et al., 2020). Therefore, AD has been largely integrated with livestock production in many parts of the world, considering it an environmentally sustainable strategy for slurry management (Bhatnagar et al., 2022). The substitution of a slurry collection system with SS involves substituting slurry in AD with the segregated faeces, referred to as the SS solid fraction (SF). Such substitution is hypothesized to improve the AD performance because of a number of SF characteristics favoring AD yield and economics. However, such comparisons are still lacking in literature.

Although slurry has many characteristics that make it a suitable substrate for AD, such as the presence of fermentative microbes facilitating the start-up of the process (Acosta et al., 2021), it also has intrinsic properties that hinder biogas production and lead to relatively low methane (CH_4) yields. These properties include a low content of OM, high ammonium nitrogen (TAN) content, and low C/N ratio (Carlsson et al., 2012; Rajagopal et al., 2013; Morken et al., 2018). Ammonium nitrogen, specifically free ammonium nitrogen (FAN), is known to inhibit the AD process at concentrations higher than 600 mg L^{-1}, especially at thermophilic temperatures (Angelidaki and Ahring, 1994; Siles et al., 2010). As for the C/N ratio, it is reported to affect substrate degradation, with ideal rates achievable within a ratio of 20–30 (Bhatnagar et al., 2022). This is higher than the C/N ratio of cattle slurry ranging from 8 to 14 (Hills, 1979; Peters et al., 2011; Neshat et al., 2017). For these reasons, dairy slurry is known to produce low specific CH_4 yield ranging from 100 to 250 L kg^{-1} volatile solids (VS) (Zhang et al., 2022; Rico et al., 2015). This results in a volumetric yield ranging from 10 to 20 L CH_4 kg^{-1} substrate, which is considered not economically viable (Angelidaki et al., 2003, Mostafa Imeni et al., 2019).

Substituting slurry with the source segregated SF leads to a lower TAN concentration and increases the C/N ratio of the feedstock, which can potentially improve the specific biogas yield (L CH_4 kg^{-1} VS. In addition, AD of the SF instead of slurry can increase the volumetric CH_4 yield (L kg^{-1} substrate) attributed to its higher VS concentration. Moreover, the digester size and transport costs may decrease in analogy to the benefits achieved with solid-liquid separation (SLS) before AD (Moller et al., 2007; Rico et al., 2012; Rico et al., 2015). The combination of AD and SLS is a common strategy in slurry management, as they provide complementary benefits in terms of increasing fertilizing value of slurry, namely by improving N/P ratio and increasing plant available N. However, previous works showed that although the liquid fractions from SLS can produce higher specific CH_4 (per volatile solids (VS)), their volumetric CH_4 yield (per kilogram of substrate) is lower than that of slurry because of their low VS content (Rico et al., 2012; Rico et al., 2015). Moreover, the solid fractions from SLS were often found to have lower specific yield than slurry (Moller et al., 2007; Rico et al., 2012). This was explained by the higher recalcitrant biomass content, namely lignocellulose, in the solid fractions of SLS as compared to slurry. The SF from SS is expected to be more adapted to AD than that from SLS, potentially because of a lower lignocellulose content. In addition, the SF from SS systems is scraped out of the animal house on an hourly basis, which means that potentially less CH_4 is lost during storage in the animal house and can thus be collected during controlled AD. The higher performance of source segregated solids in AD compared to slurry and SLS solids was confirmed for pig excreta by Vu et al. (2016). For dairy excreta however, the effect of source segregation on CH_4 production is still unknown. Investigating the performance of source segregated dairy excreta is highly relevant because there are large variations between source segregation systems used in dairy and pig housing and which influence the physical and chemical characteristics of the segregated faeces.

The SF has a DM in the range of semi-dry AD (10–20% DM) as opposed to slurry being in the range of wet AD (<10% DM). The AD parameters used for slurry should thus be adapted to SF characteristics to optimize VS degradation and to avoid downstream trade-offs in the manure management chain. Slurry is typically digested at mesophilic temperature (35–40 °C) for 20–30 days hydraulic retention time (HRT) and 2–3 kg VS day^{-1} m^{-3} organic loading rate (Wall et al., 2014). With the same HRT, the OLR of the SF would be 4–5 kg VS day^{-1} m^{-3}, which can lead to lower degradation of OM of the SF and thus to higher residual CH_4 emitted during digestate handling and storage (Ahlberg-Eliasson et al., 2021). To cope with this, SF can be digested at thermophilic temperatures (55–60 °C) allowing higher degradation of OM (Labatut et al., 2014), or diluted to below 10% DM to be digested at mesophilic 30 days HRT typically used for slurry. In terms of feeding techniques, AD can be performed in batch reactors, in fed-batch reactors, or in continuously fed (also called semi-continuously fed) reactors (Li et al., 2014). Batch AD can provide knowledge about biodegradability of the substrate, the CH_4 yield potential, and hydraulic retention time (HRT) (Li et al., 2014), but is hardly used in practice because of high inoculum requirement and process instability (Wang et al., 2021). Continuously fed reactors (often referred to as continuously stirred tank reactor (CSTR)) are more used in practice mainly because of their higher biogas production stability and economic viability (Forster-Carneiro et al., 2008; Li et al., 2014), but their utilisation in single stage for high VS content substrates, such as the source segregated SF, can lead to higher residual CH_4 and higher DM digestate (Boe and Angelidaki, 2009; Jasińska et al., 2023). The fed-batch system can offer the opportunity of higher substrate residence time, thus higher degradation, while reducing the initial inoculum required by 60% compared to batch systems (Wang et al., 2021).

In this study we aim to investigate the CH_4 yield of anaerobically digested fractions from source segregated dairy manure and to compare it with slurry. In addition, we aim at researching different approaches to digest the novel segregated solid fraction, in terms of temperature (mesophilic/ thermophilic) and feeding technique (fed-batch and continuous feeding).

2. Materials and method

2.1. Excreta collection

Samples of dairy manure fractions were collected in September 2021 from a source segregation floor and a traditional slurry pit from an experimental unit at Dairy Campus (Leeuwarden, Netherlands). The source segregation floor was a commercial system (Zeraflex) made with adjacent tiles allowing urine to drain to an underneath pit, while keeping the faeces on top. The system is equipped with an automated scraper that moves the faeces to an outside shaded storage. The traditional slurry pit and the source segregation floor were in the same experimental unit where number of cows, floor area and feeding regime were the same. The cows were Holstein breed fed with a mixed ratio consisting of grass silage, silage maize, barley meal, soybean meal, and minerals, supplemented with pelleted concentrates.

Slurry and urine were taken from ten representative points in the storage pit under the animal house using a cylinder slid through the floor slats. The ten subsamples were mixed to a homogeneous sample from which again three subsamples were taken for chemical analysis.

Segregated faeces were collected from the temporary storage which was emptied daily. From the collected faeces, three subsamples were obtained for chemical analysis. All samples were stored at 2℃ before being chemically analysed and used in the experiments.

A self-made slurry (mixture of the segregated solid and liquid fractions) was also prepared based on the mass ratio of solid to liquid after source segregation (60% solid and 40% liquid). This was done to depict biogas yield differences between the source segregated solid and slurry, regardless of the effect of their residence time in the animal house. This is because the collected slurry usually accumulates in the pit for months, which can lead to important CH_4 losses prior to use in AD.

2.2. Anaerobic digestion experiments

The different AD experiments, with temperatures; substrates; and date and location, are summarized in Table 1.

2.2.1. Biochemical methane potential (BMP) test

Batch tests were conducted at mesophilic (38°C) and thermophilic (52℃) temperatures for 90 days to determine the cumulative methane yield from the substrates following the protocol by Moset et al. (2015). The inoculum was collected from a thermophilic mono-digester of cattle manure in Aarhus University, Foulum biogas plant (Tjele, Denmark), where the experiment was conducted starting February 2023. The inoculum was then sieved to eliminate residual organic matter and then pre-incubated at (38°C) and (52℃) for a week prior to the experiment to deplete degradable organic matter. The substrates and inoculum were added to the batch bottles using a 1:1 ratio (VS based). The following substrates were used under mesophilic conditions: 10% water diluted source segregated faeces (DSF), source segregated urine (LF), collected slurry (CS), and self-made slurry (MF). The 10% dilution was selected to achieve a DM content in the DSF comparable to that of slurry (CS and MF). Under thermophilic conditions, a non-diluted source segregated faeces (SF) was used in addition to substrates used in the mesophilic test. The SF was only digested at thermophilic temperature to simulate conditions adapted for continuous feeding in practice. This because continuous feeding at 30 days HRT would results in high OLR (3.9 kg VS day^{-1} m^{-3}). To digest the SF at mesophilic temperature and 30 days HRT, the OLR can be adjusted to 2.8 kg VS day^{-1} m^{-3} (same as that of MF and CS) by diluting it to 10.8% DM. An equal HRT instead of equal OLR was considered because we aimed at maintain the same slurry loading volume and compare the yield of the two system (SS and slurry) on system basis (per kg excreta). Blank controls consisting of only inoculum were also included in both tests. All treatments were made in triplicate. The

Table 1
Summary of the anaerobic digestion experiments conducted in this study.

Experiment	Temperatures	Hydraulic retention time (HRT)/ incubation period in days	Reactor size	Substrates	Date and location
Batch test (BMP)	Mesophilic (38) Thermophilic (52)	BMP_{30}: 30 days BMP_{90}: 90 days	500 ml	Diluted solid fraction (**DSF**), collected slurry (**CS**), self-made slurry (**MF**), liquid fraction (**LF**) Solid fraction (**SF**), **DSF**, **CS**, **MF**, **LF**	February to May 2023, Aarhus University, Foulum biogas plant (Tjele, Denmark)
Continuous test	Thermophilic (52)	30	2000 ml	**SF** and **MF**	February to May 2023, Aarhus University, Foulum biogas plant (Tjele, Denmark)
Continuously stirred fed-batch	Thermophilic (55)	Average of 133	9000 ml	**SF**	July-November 2021, Environmental technology laboratory of Wageningen University and Research (Wageningen, Netherlands)

chemical characteristics of the different fractions in this test are summarized in Table 2.

The produced volume of biogas was measured and sampled on day 3, 5, 7, 10, 15, 20, 25, 30, 40, 50, 70 and 90, using a water displacement system as described in Nyang'au et al. (2023a). The CH_4 yield was then calculated and corrected to standard conditions at 273 K and 101.325kPa and expressed in term of NL kg^{-1} VS.

2.2.2. Continuously fed reactors test under thermophilic conditions

A continuous experiment was conducted under thermophilic conditions (52℃) for the SF and MF using 2 L, BPC® BioReactor Simulator continuously stirred reactors (Bioprocess Control Instruments AB, Lund Sweden) and a 30 days hydraulic retention time (HRT). Only thermophilic temperature was considered for the continuous feeding test because the batch test showed it offered faster degradation of the SF. The OLR was 2.8 kg VS day^{-1} m^{-3} 3.9 kg VS day^{-1} m^{-3} for the MF and SF, respectively. The experiment lasted for three HRTs to allow for an initial stabilizing period. The CSTRs were initially filled with inoculum obtained from a thermophilic mono-digester of cattle manure in Aarhus University, Foulum biogas plant (Tjele, Denmark), and placed in a water bath system of BPC® BioReactor Simulator to reach and maintain 52℃. After a week of incubating the inoculum for the purpose of depleting residual degradable organic matter, the two reactors were fed daily, one with the SF and one with MF. A cylinder was attached to the CSTRs inlet and used for feeding in a way limiting the introduction of oxygen.

The biogas was sampled weekly for composition analysis, and its volume was continuously measured automatically by liquid displacement and buoyancy in the gas volume-measuring device filled with acidified water, and the volume was displayed continuously using web-based software (Bioprocess Control Instruments AB, Lund Sweden). These served to calculate CH_4 yield (L kg^{-1} VS), which was then corrected to standard conditions at 273 K and 101.325kPa and expressed in term of NL kg^{-1} VS. Weekly samples were also taken from the digestates for analysis of volatile fatty acids (VFA), total ammonium nitrogen (TAN) and pH in order to monitor the stability of the AD process. Before collecting samples, the digestates were thoroughly homogenized.

2.2.3. Continuously stirred fed-batch test of the solid fraction under thermophilic conditions

Another experiment with the same solid fraction was done at LeAF facilities in Wageningen University & Research in the Netherlands (July-November 2021) using four replicates of 10 L Continuously stirred fed-batch reactors. The used fed-batch set-up was based on gradual build-up of volume in the reactors with daily feeding of the substrate without removal of an effluent. To ensure airtightness at the start of the experiment, the lids of the reactors were sealed with duct tape around the stirrers, and 5 L gas bags were attached to an outlet through a rubber stop. The O_2 content of the headspace was then monitored for the following three days to insure anaerobic conditions. An inoculum was brought from a full scale mono-digester of dairy manure in the south of the Netherlands, operating at 35 °C and 26 days retention time, and was then incubated for four weeks at 55 °C for adaptation (there was no thermophilic inoculum from a mono-digester of dairy manure available). The experiment was then initiated by loading 2 l of 2:1 inoculum to substrate VS based. After a first stabilizing phase, the feeding rate of faeces was gradually increased from 2 kg VS day^{-1} m^{-3} to 7 kg VS day^{-1} m^{-3}. The feeding was done through an inlet equipped with a rubber stop on the reactor lid. To avoid the introduction of O_2 in the headspace during feeding, the headspace was flushed with nitrogen after each consecutive feeding.

The methane percentage in the headspace was analyzed and the gas bag volume was noted every day during the first weeks, then the frequency was gradually decreased to reach once a week by the end of the experiment. After feeding, the headspace was flushed with nitrogen and sealed. The headspace and the gas bag volumes were then used to calculate the methane production. The gas samples for composition analysis were taken with a syringe and 10 cm needle to reach the center of the headspace. Gas composition analysis were then immediately done in the GC.

2.3. Analytical methods

The substrates were characterised for their content of dry matter (DM), volatile solids (VS) and total N following standard methods (APHA, 2005). DM was determined by drying the samples to a constant weight at 100 °C. Ash content was determined by loss on ignition at 550 °C for 6 h, and the VS was determined using the difference between DM and Ash content. Total N in the substrates and digestates was analysed by the Kjeldahl digestion method (Kjeltec™ 8400, Foss Analytical CO. LTD, Denmark). Total volatile fatty acids (VFA) were determined in an oxalic acid filtrate obtained by extracting 1 g of the substrate (Feng et al., 2018). VFA concentration and acid profile (acetic, propionic, butyric, and valeric acids) were determined by 7890 A gas chromatography equipped with a flame ionisation detector and helium gas as a carrier gas (Agilent Technologies, CA 95051, USA). The temperatures of the initial oven,

Table 2
Chemical properties of the dairy manure fractions and slurry. The values correspond to mean± Std (n=3). The letters indicate significant differences. Means with different letters within each column are significantly different ($p < 0.05$).

	DM	VS	Total-N	TAN	TAN/ Total-N	Total-P	pH
	%	%	g L^{-1}	g L^{-1}	%	g kg^{-1}	
SF	14.0^{a}±0.1	11.7^{a}±0.1	4.2^{b}±0.0	0.9^{d}±0.1	21^{d}±0.1	1.09^{a}±0.00	6.7^{c}±0.1
DSF	10.8^{b}±0.1	9.1^{b}±0.1	3.8^{d}±0.0	0.8^{d}±0.1	21^{d}±0.1	0.97^{b}±0.00	6.7^{c} ±0.1
LF	3.0^{c}±0.3	1.2^{d}±0.2	3.7^{d}±0.1	3.4^{a}±0.0	92^{a}±0.1	0.04^{d}±0.00	9.1^{a}±0.0
CS	10.4^{b}±0.9	8.2^{c}±0.1	4.5^{a}±0.0	2.3^{b}±0.0	51^{b}±0.1	0.65^{c}±0.00	7.2^{b}±0.1
MF	10.7^{b}±0.1	8.4^{c}±0.1	4.0^{c}±0.1	1.7^{c}±0.1	43^{c}±0.1	0.71^{c}±0.00	7.3^{b}±0.1

DM: dry matter, VS: Volatile solids, TAN: total ammoniacal nitrogen injector port and detector were 100°C, 285°C and 300°C respectively. Total ammonium nitrogen (TAN) concentration in the substrates, weekly digestate samples, and in the final digestates was determined using photometry (Spectroquant Kit, Merk, NJ 07033, USA).

Fibre analysis entailed drying the samples at 60 °C for 48 h and grinding them to 0.8 mm particle size using Cyclotec™ 1093 mill (Foss, MN 55344, USA). The prepared samples were then used to measure acid detergent fibre (ADF), acid detergent lignin (ADL) and neutral detergent fibre (NDF) using a Foss Fibertec 2010 System (Foss Analytical CO. LTD, Denmark), to determine the hemicellulose, cellulose, lignin contents as described by Van Soest et al. (1991). Hemicellulose was calculated as the difference between NDF and ADF, cellulose as the difference between ADF and ADL, and lignin content was assumed to be equivalent to ADL. Total carbon content was analysed by Vario MAX cube elemental analyser (Elementar Analysensysteme GmbH,Germany). Lipids were analyzed according to Jensen (2008), while proteins were estimated using a multiplication factor according to Hayes (2020):

$$\text{Protein content (g kg}^{-1}) = (\text{Total N} - \text{TAN}) \times 6.25 \tag{1}$$

The gas composition was analysed using 7890 A gas chromatography (Agilent technologies, CA 95051, USA) coupled with a thermal conductivity detector and helium as carrier gas. The measured CH_4 content was corrected as a proportion of biogas's CH_4 + CO_2 contents. The specific methane yields were normalised to standard conditions (273 K and 101.325 kPa).

2.4. Calculations

2.4.1. Fed batch test methane yield

During the experiment, we noticed that the volume of the gas bags attached to the CSTRs was not increasing because the pressure build-up in the headspace was too low given its initial volume (8 l). Thus, to estimate the methane volume build-up, the methane percentage increase in time in the headspace was followed. The methane percentage was found to increase and stabilize after around 48 h, in a range of 50–60%. This information was then used to estimate methane volume during the period where gas composition was not analyzed daily. When gas composition was measured daily, the methane volume was calculated using Eq. 3. When it was not measured daily, Equation 4 was used:

$$MV_n\ (l) = V_n \times M_n\% \tag{2}$$

$$MV_n\ (l) = (D/2) \times V_n \times M_n\% \tag{3}$$

where, MV_n was the CH_4 volume produced between measurement n-1 and measurement n, $M_{n\%}$ the percentage of CH_4 at measurement n, V_n the volume of the headspace at measurement n, and D the number of days between n and n-1 measurements (considering the $M_{n\%}$ needed 48 h to stabilize when the headspace is saturated).

Table 3
Biochemical methane potential (BMP) from mesophilic and thermophilic batch experiments with the manure fractions and slurry. The values correspond to mean ± Std (n=3). The letters indicate significant differences. Means with different letters within each column are significantly different ($p < 0.05$).

BMP_{30} (NL kg^{-1} VS)	Mesophilic	Thermophilic
SF	-	213 ± 4^{b}
DSF	208± 2^{b}	229± 1^{a}
CS	153± 5^{d}	183± 11^{c}
MF	192± 4^{c}	209± 4^{b}
LF	654± 21^{a}	0± 0*
Volumetric yield$_{30}$ (NL kg^{-1} substrate)		
SF	-	24.9± 0.5
DSF	18.9± 0.2	20.8± 0.1
CS	12.5± 0.4	15.0± 0.9
MF	16.1± 0.3	17.5± 0.3
LF	6.7± 0.2	0±0*
Weighted volumetric yield$_{30}$ (NL kg^{-1} excreta**)		
SF	-	14.9± 0.5
DSF	12.5± 0.2	13.7± 0.1
CS	12.5± 0.4	15.0± 0.9
MF	16.1± 0.3	17.5± 0.3
LF	2.7± 0.2	0±0*

(-)not tested
(*) prosses failed
(**) calculated by multiplying the yield/ kg substrate by 60% for the solid fraction (represents 60% of the excreta), 66% for the DSF, 40% for the LF, and 100% for CS and MF.

2.5. Statistical analysis

The differences in physical and chemical properties and methane yields between fractions in the batch test were statistically tested using a General linear model performed in SPSS 28 (IBM Corp, 2021). The independence of variables and normality of residual observations were checked prior to fitting the models. When significant differences were obtained, the equality of variance between groups was checked using Levene's test. For multiple comparisons of means, Tukey B test was used when the variances were found equal, while Dunnett's T3 test was used when the variances were found non-equal.

3. Results and discussion

3.1. Biochemical methane potential test

The biochemical methane potential at 30 days (BMP_{30}) and the corresponding CH_4 volumetric yield of the SF, DSF, MF, CS and LF are summarized in Table 3. The BMP_{30} of the LF is only shown under mesophilic conditions because no biogas was produced from this fraction under thermophilic conditions. This was hypothesized to be related to possible methanogens inhibition caused by the high TAN content and pH of the LF (Yenigün and Demirel, 2013).

The results showed that BMP_{30} ranged from 153 to 229 NL kg^{-1} VS for the solid fractions and slurry, which is in line with previous studies for dairy manure CH_4 yields typically ranging from 100 to 250 NL kg^{-1} VS (Rico et al., 2012; Zhang et al., 2022). The LF had the highest BMP_{30} with 654 NL kg^{-1}VS, which also was in the range of previous findings related to liquid fractions from slurry separation (Rico et al., 2012). A higher specific CH_4 yield of separated or segregated liquid fractions could be explained by their VS content being composed mainly of highly degradable material (Møller et al., 2007; Rico et al., 2012; Vu et al., 2016). Under mesophilic conditions, the DSF yielded 208 NL kg^{-1}VS, which was significantly higher than the 192 and 153 NL CH_4 kg^{-1}VS yielded by the MF and CS respectively. The difference between the specific yield of CS and MF could be due to storage time and conditions, as Browne et al. (2015) found that starting from 8 weeks of storage at 20℃, significant reduction in slurry AD yield can be observed. Under thermophilic conditions, DSF again yielded significantly higher specific CH_4 (229 NL kg^{-1}VS) compared to MF (209 NL kg^{-1}VS), CS (183 NL kg^{-1}VS) and SF (213 NL kg^{-1}VS) (Table 3). The difference observed between DSF and SF yields could be related to the findings of Jeppu et al. (2022), where water dilution of slurry increased AD yield by possibly improving mass transfer and viscous mixing. The SF and MF specific yields were comparable (not significantly different at $p<0.05$). It is expected that SF produces lower specific CH_4 yield than MF because of its possible lower content of more easily degradable VS, in analogy with the solid fractions from solid-liquid separation of slurry. For example, Rico et al. (2012) reported 21% higher specific CH_4 yield in slurry compared to the solid fraction from SLS (by flocculation) of slurry. This difference in yield between SF and MF was not observed in our case probably because faeces (the solid fraction of source segregation) were different in chemical characteristics than the solid product of SLS (Vu et al., 2016). For instance, the greatest difference between them is lower DM, VS and possibly lower fiber content in the source segregated faeces. Further, even with higher concentration of easily degradable VS, and thus higher CH_4 potential, the MF might have had a reduction of its BMP potential under thermophilic conditions due to TAN inhibition. This is because we observed a failure of the AD process with the LF (constituting 40% of the MF volume). Vu et al. (2016) also found that source segregation solids yielded higher specific CH_4 compared to the solids from four types of SLS tested in the study.

Volumetric CH_4 yield calculations showed that the SF under thermophilic conditions outperformed all the tested substrates with 24.9 NL CH_4 kg^{-1} substrate, compared to 20.8, 15.0 and 17.5 NL CH_4 $kg^{-1}$for DSF, CS and MF, respectively under thermophilic conditions, and 18.9, 12.5, and 16.1 NL CH_4 $kg^{-1}$for DSF, CS and MF, respectively under mesophilic conditions (Table 3). These yields are in agreement with previous studies, as cattle manures are typically reported to yield from 10 to 20 l CH_4 kg^{-1} (Angelidaki et al., 2003; Rico et al., 2012; Liebetrau et al., 2021).

When considering the mass separation (1 kg of CS or MF is equivalent to 0.6 kg of SF, 0.66 kg of DSF and 0.4 kg of LF estimated by Galama et al. (2021)), under thermophilic conditions the SF produced 14.9 NL of CH_4 per 1 kg of excreta, which represents 99% of the

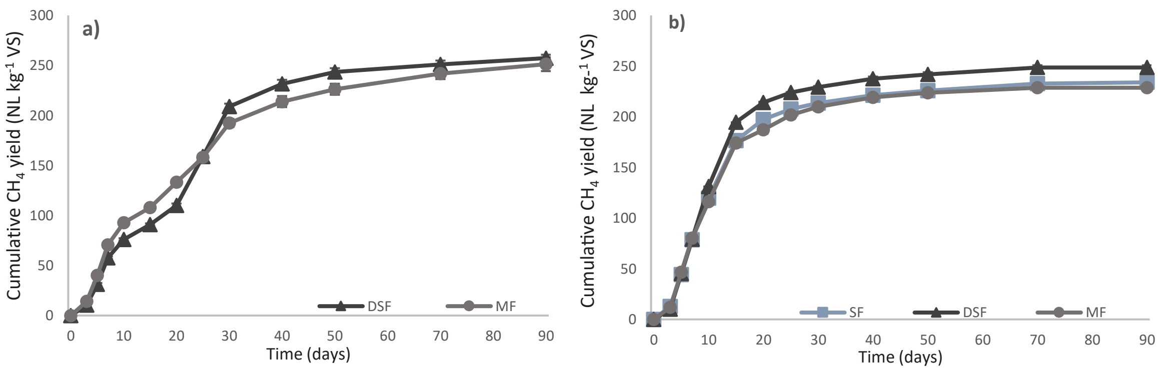

Fig. 1. Development of the cumulative CH_4 yield (NL/ kg VS) from the BMP of source segregation fractions and slurry under mesophilic (a) and thermophilic (b) conditions. Error bars represent standard deviation (n=3).

CS production (15.0 NL kg^{-1} excreta) and 85% of the MF production (17.5 NL kg^{-1} excreta). This means that at least 85% of total CH_4 in the excreta derived from the SF, constituting only 60% of the mass. This could reduce the reactor size by 40% compared to slurry digestion. Under mesophilic conditions, the DSF produced 12.5 NL CH_4 kg^{-1} excreta, which represents 100% of CS production (12.5 NL kg^{-1} excreta) and 78% of MF production (16.1 NL kg^{-1} excreta).

The ultimate CH_4 yield (BMP_{90}) was also followed for DSF, SF, and MF, as shown in Fig. 1. The results showed that after 90 days the yields reached 234, 249, and 229 NL CH_4 kg^{-1}VS for SF, DSF and MF, respectively under thermophilic conditions, and 257 and 251 NL CH_4 kg^{-1}VS for DSF and MF, respectively under mesophilic conditions. A higher specific yield in mesophilic compared to thermophilic temperature was also found by Wang et al. (2014) in manure substrates with C/N ratio lower than 25. However, in our results all the substrates reached the 200 NL CH_4 kg^{-1}VS in 25 days under thermophilic conditions, while this value was only reached after 30 days under mesophilic conditions. This suggested that thermophilic conditions results in faster degradation of VS and can eventually reduce the required HRT under continuous feeding, which is also in line with findings of Liu et al. (2022). The advantage of faster VS degradation under thermophilic conditions is even more relevant in the case of the SF because of its higher VS. Poor degradation of substrates during AD could lead to higher residual CH_4 emissions from the digestate during storage and increased risk of NH_3 volatilization following field application (Nyang'au et al., 2023b). Diluting the SF prior to mesophilic digestion also seems an effective approach to improve degradation as DSF reached significantly higher specific CH_4 yield than MF at 30 days. However, this approach would compromise the advantage of reduced volume compared to slurry. Thermophilic digestion seems thus a more viable option under continuous feeding of the SF in order to improve degradation rate and reduce the digester volume.

3.2. *Thermophilic CSTR test*

3.2.1. *Effect on manure fractions chemical characteristics*

The chemical properties of the SF and MF and their respective digestates are summarized in Table 4. The results showed that the AD process decreased the DM content of the SF from 14.0% to 11.5%, and that of the MF from 10.7% to 8.3%. These reductions correspond to 18% and 22% relative reductions for SF and MF, respectively, which are similar to the 18.6% found in Möller et al. (2008). The total carbon content was also reduced from 61.5 to 39.0 g kg^{-1} FM for the SF, and from 33.8 to 29.5 g kg^{-1} for the MF, meaning that higher C degradation occurred during AD of the SF. This could be related to the higher initial C/N ratio in the SF (14.6) compared to the MF (8.5). In general, anaerobic microbes utilize carbon 25–30 times faster than nitrogen, which is why an optimal C/N ratio for carbon degradation to biogas is defined to be in the range of 25–30 (Khanal et al., 2019). For substrates like cattle slurry containing poorly degradable material, such as lignin, the optimal C/N ratio is in the range of 16–19 (Mshandete et al., 2004). Although the C/N ratio of both SF and MF was not in the optimal range, 14.6 can already result in higher C degradation than 8.5, as Hills (1979) found that increasing the C/N ratio of dairy manure from 8 to 11.2 resulted in 26% increase in biogas production. The C reduction ultimately reduced the C/N ratio, which reached 9.7 and 7 for the SFD and MFD, respectively.

The TAN/ TN ratio was also altered for both fractions following AD, increasing from 22% to 45% DM (100% increase) for the SF and from 42% to 61% DM (47% increase) for the MF. Such increase is explained by organic N mineralization following organic matter degradation during the AD process (Mortensen J. et al., 2021). The results suggest that higher N mineralization occurred in the SF as compared to the MF, which is consistent with the higher C degradation in the SF. These findings are consistent with other studies who found an increase in TAN/ TN in the range of 6–53%, with variation depending on slurry characteristics, AD process parameters and pre-treatments (Westerholm et al., 2016; Nyang'au et al., 2022; Wilińska-Lisowska et al., 2023). The MF results fall under this range but the SF ones are considerably higher. This could be related to the much lower initial TAN/TN of the SF compared to slurry and to its higher content of organically bound N.

3.2.2. *Effect on CH_4 yield*

The CSTR tests was continued for 12 weeks corresponding to a 2.5 HRTs, being 76 days, to allow for process stabilization during which the daily specific CH_4 yield, the total VFA concentration, individual VFA profiles, the TAN concentration, and the pH were monitored weekly. The results are shown in Fig. 2(a. Daily specific CH_4, b. Total VFA concentrations, c. pH and d. FAN concentration).

The daily specific CH_4 yield calculations showed that the MF had a more rapid yield increase compared to the SF until day 35, after which the SF consistently produced higher CH_4 reaching 205 NL CH_4 kg^{-1} VS compared to 140 NL CH_4 kg^{-1} VS for the MF by the end of the experiment. The average daily yields were 174 and 105 NL CH_4 Kg^{-1} VS for SF and MF, respectively, after elimination of the yields of the first HRT considering it was a start-up phase. These yields were much lower than those of the BMP test at 30 days (213 and 209

Table 4
Chemical properties of the substrates and digestates from anaerobic digestion of solid fraction (SF) and self-made slurry (MF) under thermophilic temperatures and in a continuous stirred tank reactor (CSTR). The values correspond to mean± Std (n=3).

	DM	VS	Total-N	TAN	TAN/Total-N	Organic-N	C	C/N
	%	% DM	g Kg^{-1} DM	g Kg^{-1} DM	%	g Kg^{-1} DM	g Kg^{-1} FM	-
SF	14.0±0.1	84.1±0.1	30.4±0.0	6.8±0.1	22±0	23.6±0.0	61.5±1.2	14.6±0.1
SF digestate	11.5±0.2	81.9±0.2	37.2±0.0	16.6±0.1	45±0	20.6±0.0	39.0±1.7	9.1±0.1
MF	10.7±0.1	78.4±0.1	37.8±0.0	15.8±0.0	42±0	22.0±0.0	33.8±0.3	8.5±0.0
MF digestate	8.3±0.0	71.3±0.0	50.0±0.0	31.1±0.1	62±0	19.5±0.0	29.5±0.5	7±0.0

DM: dry matter, VS: Volatile solids, TAN: total ammoniacal nitrogen.

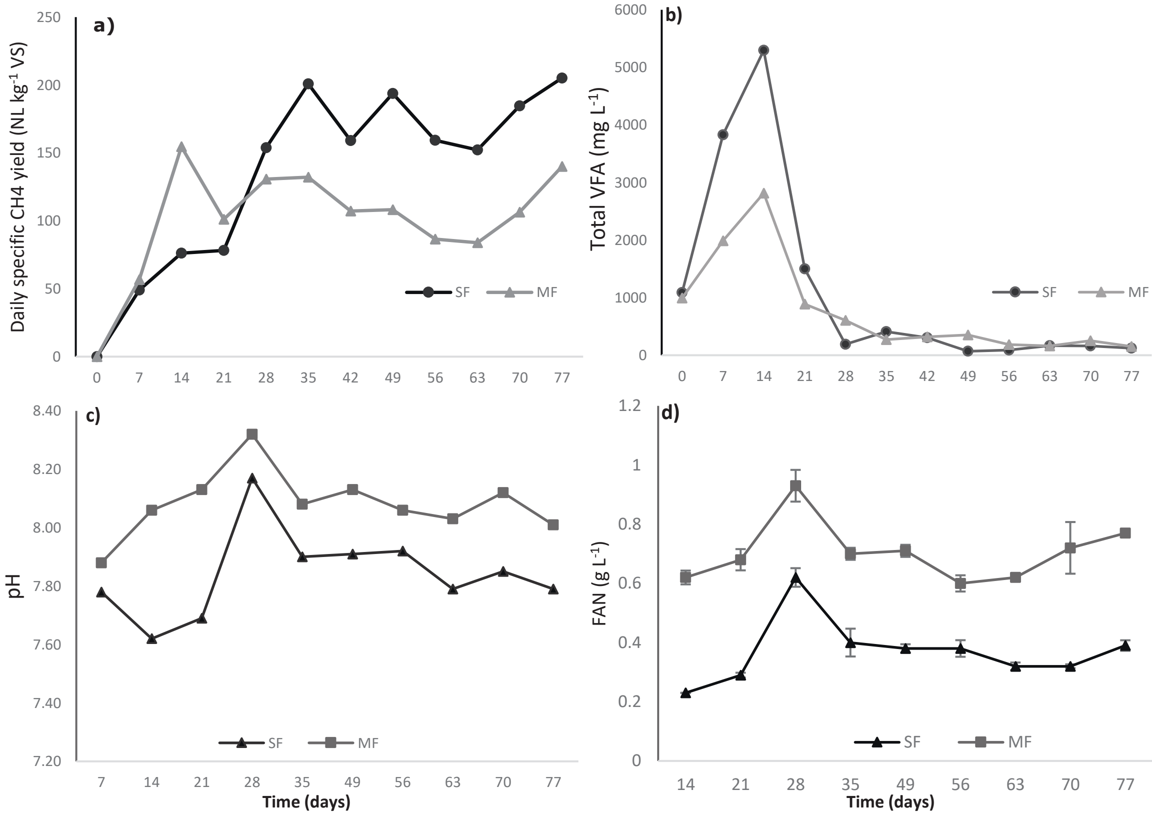

Fig. 2. Development of the daily CH_4 yield (a) and total VFA (b), pH (c) and FAN concentration (d) during the CSTR test with the solid fraction and self-made slurry. Error bars in FAN figure represent standard deviation of n=3 measurement replications.

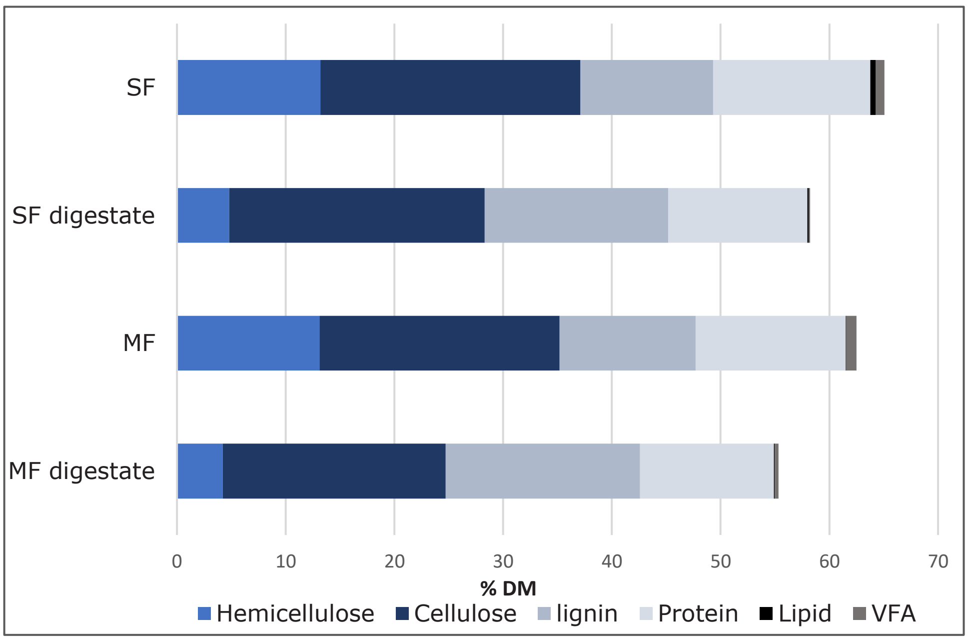

Fig. 3. Fibre, protein, lipid and VFA composition of the solid fraction (SF) and self-made slurry (MF) and their respective digestates (SFD and MFD) from the CSTR test.

NL CH_4 kg^{-1} VS for SF and MF, respectively). Li et al. (2014) also found higher CH_4 yield with the batch set-up compared to the CSTR using chicken manure as a substrate. The higher performance of the batch reactors can be explained by quicker process adaptation, especially to high ammonia concentrations when using an inoculum adapted to such conditions. In our case the inoculum used came from a full scale thermophilic reactor co-digesting cattle manure.

The initial low CH_4 yield of the SF can be related to the observed accumulation of total VFA reaching over 5000 mg L^{-1} (compared to 2816 mg L^{-1} for the MF) (Fig. 2.b). A total VFA concentration of over 6000 mg L^{-1} can inhibit methanogens and thus CH_4 production (Ajayi-Banji et al., 2022). Park et al. (2018) reported that individual VFA profiles, and specifically the propionic acid to acetic acid ratio, are a better indicator of VFA toxicity to methanogens because a high acetic acid concentration accompanied with H_2 accumulation inhibits propionic acid degradation. A propionic acid to acetic acid ratio of over 1.4 was defined to be alarming (Hill et al., 1987), which was exceeded for the SF on day 21 being 2.5 (Fig. 3 in Supplementary material), following which we increased the stirring speed for both reactors to improve degradation and suppress accumulation of H_2. This intervention reduced the total VFA and propionic acid/ acetic ratio for the SF to 0.1, which was followed by an increase in CH_4 production for the SF (Fig. 2.a). The MF yield was however not affected.

The lower CH_4 yield of the MF compared to SF in the CSTR was not observed in the batch test (Table 3), which could mean that SF adapted better than the MF to the continuous feeding set-up, possibly because of the higher initial TAN concentration in the MF. Ammonia toxicity to the AD process can be assessed by the free ammonia (FAN) concentration that is reported to be inhibitory for cattle manure substrates when exceeding 600 mg L^{-1} at thermophilic conditions (Angelidaki and Ahring, 1994; Siles et al., 2010). FAN concentration during the experiment was on average 700 mg L^{-1} for the MF and 330 mg L^{-1} for the SF, and reached a maximum of 930 mg L^{-1} for MF and 620 mg L^{-1} for SF (Fig. 2.d). Therefore, FAN was at inhibitory concentrations during the whole experiment period for the MF, contrarily to the SF. This can confirm the poorer process stability with the MF substrate and thus explain its lower CH_4 yield. Another parameter that can influence ammonia toxicity is the initial C/N ratio of the substrate, as a low initial C/N ratio was found to lower the inhibition threshold at thermophilic temperatures (Jiang et al., 2019). The C/N ratio also influences the C degradation during AD as referred to in Section 3.1. The C degradation results showed accordance with the CH_4 yield results, considering that the higher C degraded from the SF corresponds to C recovered as biogas (CH_4+CO_2), thus explaining the higher yield of this fraction.

Finally, the volumetric CH_4 yield calculations summarized in Table 5 showed that the SF and MF yielded 12.2 and 9.9 NL CH_4 kg^{-1} excreta, respectively, meaning 23% higher CH_4 was produced when using only the segregated solid fraction although the increase was not statistically significant ($p<0.05$).

3.2.3. Effect on volatile solids components degradation

The characterization of fiber (hemicellulose, cellulose and lignin), protein, lipid and VFA in the substrates and digestates are shown in Fig. 3. For fiber, the results showed that the SF and MF had little initial difference. The lignin content was 12.2 and 12.5% of DM, the cellulose 23.9 and 22.1% of DM and hemicellulose 13.2 and 13.1% of DM for SF and MF, respectively, which were in line with Møller et al. (2014) and Cu et al. (2015). The results showed that source segregation did not create a different fraction compared to slurry when it comes to lignocellulose composition, contrarily to what is reported for solid-liquid separation techniques such as centrifugation. Separating slurry using centrifugation transfers most of the recalcitrant matter (lignin) to the solid fraction.

The estimation of protein content showed that the SF and MF had 14.5 and 13.9% DM, respectively. This is in accordance with the range reported by Møller et al. (2014). Further, lipid characterization showed that both substrates had a low content but with significantly higher one ($p<0.01$) for the SF (0.9% compared to MF, 0.1% DM). Our lipid contents are lower than in Moset et al. (2015) and Cu et al. (2015) who found between 2.3% and 6.4% DM. This could be related to a difference in feed composition (Møller et al., 2014). The VFA content was also found low for both substrates, with 0.8 and 0.9% for SF and MF, respectively which is comparable to Møller et al. (2014).

The degradation of fiber, protein, lipid and VFA during AD were calculated and presented in Table 6. The results showed that the neutral detergent fiber (NDF) per fresh matter was reduced by 25% and 31% for SF and MF, respectively corresponding to 16.9 and 15.5 g kg^{-1} FM. Hemicellulose had the highest degradation with 70 and 75% reduction for the SF and MF respectively, followed by cellulose with 19 and 28% reduction for SF and MF, respectively. The lignin content per fresh matter slightly increased for both fractions (14% for SF and 11% for MF) after AD, which could be explained by the difference in fresh weight between the substrates and digestates due to biogas production and water vapor loss. Nyang'au et al. (2022) also found a slight increase in lignin content per fresh matter after substrates AD, which they attributed to enrichment of the lignin in the digester. Moreover, lignin is known to be the recalcitrant part of the lignocellulosic matter and thus will often not be degraded during AD (Triolo et al., 2011). There were no

Table 5
Specific and volumetric CH_4 yields of the solid and self-made slurry. The values correspond to mean± Std (n=3). The letters indicate significant differences. Means with different letters within each raw are significantly different ($p<0.05$).

	Solid fraction	Self-made slurry
Average specific yield in the last HRT (NL CH_4 kg^{-1} VS)	$174.0^a\pm25.3$	$105.0^b\pm20.3$
Average volumetric yield in the last HRT (NL CH_4 kg^{-1} substrate)	$20.3^a\pm3.0$	$9.9^b\pm1.9$
Average volumetric yield in the last HRT (NL CH_4 kg^{-1} excreta*)	$12.2^a\pm1.8$	$9.9^a\pm1.9$

* Calculated by multiplying the yield/ kg substrate by 60% for the solid fraction (represents 60% of the excreta) and 100% for slurry (represents 100% of the excreta). HRT: hydraulic retention time.

Table 6
Fibre, protein, lipid and VFA degradation in the solid fraction and self-made slurry after and their respective digestates. The values represent mean or mean ± std (n=3). The letters indicate significant differences between degradation of SF and MF: means with different letters within each column are significantly different (p <0.05).

		Hemicellulose	Cellulose	Lignin	NDF	Protein	Lipid	VFA
SF	Substrate	18.4	33.4	17.0	68.9	20.2±0.5	0.7±0.0	1.1±0.0
g/kg FM	Digestate	5.5	27.0	19.4	51.9	14.7±0.1	0.2±0.0	0.1±0.0
	Degradation g/ kg FM	12.93	6.39	-2.37	16.9	5.5±0.4[a]	0.6±0.0[a]	1.0±0.1[a]
	(%)	(70%)	(19%)	(-14%)	(25%)	(27%)	(78%)	(83%)
MF	Substrate	14.0	23.6	13.3	50.8	14.4±0.4	0.1±0.0	1.0±0.0
g/kg FM	Digestate	3.5	17.0	14.9	35.3	10.2±0.7	0.1±0.0	0.3±0.0
	Degradation g/ kg FM	10.5	6.6	-1.5	15.5	4.3±0.5[b]	0.0±0.0[b]	0.7±0.1[b]
	(%)	(75%)	(28%)	(-11%)	(31%)	(29%)	(0%)	(72%)

SF: solid fraction, MF: Self-made slurry, NDF: neutral detergent fiber, VFA: volatile fatty acids.

replications of measurements because of lack of samples, thus, a statistical significance of the differences between fiber degradation of MF and SF could not be concluded.

For protein, the results showed that 27% and 29% FM of protein was degraded during AD of the SF and MF, respectively, corresponding to 5.5 and 4.3 g kg^{-1} that were significantly different ($p<0.05$). Protein is known to produce around 0.6 L CH_4 g^{-1} (Alves et al., 2009) meaning that the higher protein mass degraded in the SF compared to the MF could partly explain the higher CH_4 yield of the SF. In addition, the SF also had 78% lipid degradation (0.6 g kg^{-1}) while the MF showed no lipid degradation related to the very low initial content. Lipids produce around 1.0 L CH_4 g^{-1} (Alves et al., 2009), meaning a higher lipid degradation in the SF could contribute to its measured higher CH_4 yield. Finally, the results showed that VFA degradation was 89 and 72% for SF and MF respectively, corresponding to 1.0 and 0.7 g kg^{-1} FM.

3.3. Fed-batch anaerobic digestion test

3.3.1. Dry matter and volatile solids reduction

The results showed that the fed-batch AD reduced the DM content of the solid fraction by 35% (from 15.0% to 9.7%), and the VS by 8% (85.8–78.8% DM). These reductions were higher than what we found with the CSTRs test, which can be explained by the higher residence time of the substrate (133 days on average compared to 30 days in CSTR test).

3.3.2. Effect on methane yield

The fed batch test was started with an adaptation phase where feeding was gradually increased from 2 to 7 g COD m^{-3} day. During this phase the volatile fatty acids and pH were monitored and used to adapt feeding rate. After 30 days, the reactors ran continuously without instability following the increase of feeding rate. This can be seen from the CH_4 percentage in the produced biogas after day 30 (Fig. 4.b), which increased rapidly to reach stable values between 50% and 60% during the rest of the experiment for the four reactors.

Fig. 4.a displays the development of the cumulative specific CH_4 yield, and shows that by 80 days the SF yielded 356 NL CH_4 kg^{-1} VS corresponding to 24.5 NL CH_4 kg^{-1} excreta, calculated as a mean from four reactors. This was 55% higher than the yield obtained in the batch test at 90 days. Wang et al. (2021) also found 45% higher cumulative specific yield in the fed-batch set-up compared to a the batch set-up when digesting a solid substrate of cattle manure, cornstalk and tomato residues. The authors explained the difference mainly by the higher residence time of substrates in the fed-batch reactor (55 days more than the batch reactor), which allows more

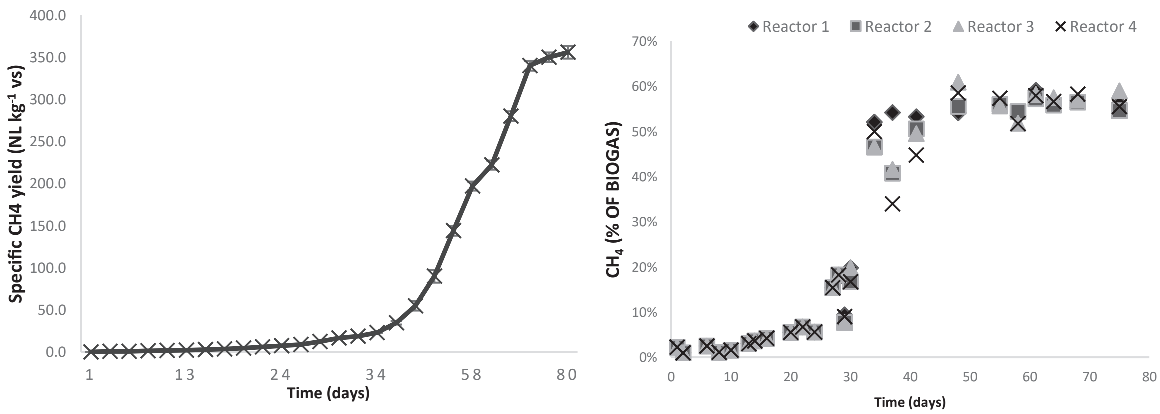

Fig. 4. Development of the cumulative CH_4 yield (a) and CH_4 percent in the biogas (b) during the fed-batch test with the solid fraction. The error bars in (a) are based on four replicates.

time for organic matter degradation. Our results could also be explained by the 45 days higher residence time in the fed-batch reactor. In the latter substrates residence time was on average 133 days.

3.4. Perspectives

The results of this study demonstrated that substituting slurry collection systems with source segregation systems offers advantages for anaerobic digestion of the excreta. Most importantly, the solid fraction had significantly higher CH_4 yield in the BMP test as compared to slurry collected from the pit, but not compared to self-made slurry. This was attributed to the collected slurry having possibly lost considerable CH_4 during temporal storage in the animal house, meaning that source segregation can increase CH_4 yield of dairy excreta by reducing losses prior to controlled AD. Further, higher CH_4 yield was obtained with the solid fraction as compared to the self-made slurry in the CSTR set-up. We found that this higher yield could mainly be attributed to the lower initial TAN concentration and higher C/N ratio of the source segregated solid fraction compared to slurry. These results are different compared to what is reported with solid-liquid separation of slurry, where the SF produced lower specific CH_4 yield compared to slurry. Møller et al. (2007) and Rico et al. (2012) explained this lower yield by the fact that solid-liquid separation concentrates most of the easily degradable matter in the liquid fraction and recalcitrant matter in the solid fraction. This could be the main difference with source segregated solid fraction, which we found had similar lignocellulosic profile to slurry. Another difference between the separation systems is source segregation being able to reduce CH_4 losses prior to controlled AD, contrarily to solid-liquid separation performed after temporal storage of slurry in the pit. Moreover, in addition to not compromising the CH_4 yield, substituting slurry with the source segregated solid fraction in anaerobic digestion can allow a 40% reduction in digester size and possibly reduced transport cost. This can increase the economic feasibility of mono-digestion of dairy manure. However, further research should follow to verify and quantify possible economic gains with this substitution.

Other benefits of combining anaerobic digestion to source segregation include improving availability of nutrients and balance in the manure. The TAN/TN in the solid fraction increased from 22% to 45% after anaerobic digestion which will increase its N fertilizing value after soil application. In addition, the source segregated solid contains most of the P from dairy excreta, while the liquid fraction has an N/P ratio of more than 90. This can improve the N:P balance during fertilization with dairy manure compared to fertilizing with slurry digestate. Further research is however required to quantify the NH_3 and CH_4 emissions from the storage and soil application of the novel solid fraction digestate.

In this study we also found that fed-batch digestion produced a 46% higher cumulative yield than the continuously fed reactors. In addition, the fed-batch reduced the dry matter content more effectively, and resulted in a SF digestate with 9.7% dry matter compared to 11.5% with the continuously fed reactors. Higher dry matter content in the digestate can lead to higher residual CH_4 when stored and higher NH_3 emissions when soil applied because of the low infiltration into the soil (Sommer and Olesen, 1991). Thus, a fed-batch set-up could be more beneficial for substrates with high dry matter, such as the source segregated solid faeces, because higher yield is obtained and a lower emission digestate could potentially be produced.

4. Conclusion

This study set out to unveil the methane yield of source segregated dairy faeces (solid fraction) and urine (liquid fraction) in comparison to that of slurry, on system basis (per kg excreta), and to explore different anerobic digestion approaches to digest the novel segregated solid fraction. We found that the specific CH_4 yield (BMP_{30}) by thermophilic and mesophilic AD of source segregated SF were significantly higher than that of slurry collected from the pit, but comparable (not significantly different at $p<0.05$) to that of a self-made slurry (by mixing faeces and urine). This highlighted that source segregation allows to increase CH_4 yield of dairy excreta by possibly reducing storage time and thus limiting CH_4 losses in the animal house. Although the BMP_{90} was higher under mesophilic temperatures, under thermophilic temperature a faster production rate was observed resulting in higher BMP_{30} for all substrates. The CSTR test at 30 days HRT was thus conducted at thermophilic temperature and showed that the SS solid fraction had higher C degradation, higher N mineralization and produced 20% higher CH_4 kg^{-1} excreta as compared to the self-made slurry. We concluded that substituting slurry with the source segregated solid fraction is anaerobic digestion can produce similar or higher CH_4 per kg excreta with solid fraction representing only 60% of the excreted volume vs slurry, which can increase economic viability of dairy excreta AD. The fed-batch allowed 45% higher CH_4 yield than the CSTRs for the SF and higher DM degradation, which can further reduce CH_4 and NH_3 emissions downstream. Further investigations of downstream management of the digestate is needed to prove this.

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CRediT authorship contribution statement

Jerke De Vries: Writing – review & editing, Supervision, Methodology, Conceptualization. **Jihane EL Mahdi:** Writing – review & editing, Writing – original draft, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Jared Onyango Nyang'au:** Writing – review & editing, Methodology, Investigation. **Henrik Bjarne Møller:** Writing – review & editing, Validation, Supervision, Resources, Methodology, Conceptualization. **Miriam H.A. Van Eekert:** Writing – review & editing, Supervision, Methodology, Conceptualization. **Peter Groot Koerkamp:** Writing – review & editing, Supervision, Resources, Project administration, Funding acquisition, Conceptualization.

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.

Data availability

A link to the data file will be provided with the online article after manuscript acceptance

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.eti.2024.103684.

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