

Dry anaerobic digestion of high solids content dairy manure at high organic loading rates in psychrophilic sequence batch reactor

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Received: 13 January 2015 / Revised: 27 February 2015 / Accepted: 1 March 2015 / Published online: 14 March 2015
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Abstract Cow manure with bedding is renewable organic biomass available around the year on dairy farms. Developing efficient and cost-effective psychrophilic dry anaerobic digestion (PDAD) processes could contribute to solving farm-related environmental, energy, and manure management problems in cold-climate regions. This study was to increase the organic loading rate (OLR), fed to a novel psychrophilic (20 °C) dry anaerobic digestion of 27 % total solid dairy manure (cow feces and wheat straw) in sequence batch reactor (PDAD-SBR), by 133 to 160 %. The PDAD-SBR process operated at treatment cycle length of 21 days and OLR of 7.0 and 8.0 g total chemical oxygen demand (TCOD)kg⁻¹ inoculum day⁻¹ (5.2±0.1 and 5.8±0.0 g volatile solids (VS)kg⁻¹ inoculum day⁻¹) for four successive cycles (84 days) produced average specific methane yields (SMYs) of 147.1±17.2 and 143.2±11.7 normalized liters (NL)CH₄kg⁻¹ VS fed, respectively. PDAD of cow feces and wheat straw is possible with VS-based inoculum-to-substrate ratio of 1.45 at OLR of 8.0 g TCOD kg⁻¹ inoculum day⁻¹. Hydrolysis was the limiting step reaction. The VS removal averaged around 57.4±0.5 and 60.5±5.7 % at OLR 7.0 and 8.0 g TCOD kg⁻¹ inoculum day⁻¹, respectively.

Keywords Anaerobic digestion · Cow manure · Dry · Psychrophilic · Wheat straw · High rate

Electronic supplementary material The online version of this article (doi:10.1007/s00253-015-6516-2) contains supplementary material, which is available to authorized users.

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Introduction

Dry anaerobic digestion is increasingly attracting the interest of farmers to digest farm waste and agriculture residue. Cabral et al. (2014) indicated that there are currently more than 200 plants of dry anaerobic digestion worldwide to digest organic material with total solids (TS; Table 1) ranging between 20 and 50 %, mostly in thermophilic operation. In this respect, livestock industry, which produces a large amount of manure requiring treatment and stabilization, can benefit from dry anaerobic digestion. In Canada and USA, cattle generate about 75 and 86 % of the manure produced by livestock, respectively (Hofmann and Beaulieu 2001; Wen et al. 2004). Dairy cow feces and bedding mixtures are composed of 40–50 % biodegradable lignocellulosic biomass which can be converted to CH₄ (Abbassi-Guendouz et al. 2012).

Dry anaerobic digestion (DAD) became the process of choice to treat the organic fraction of the municipal solid waste (Cabral et al. 2014; Challen Urbanic et al. 2011; Li et al. 2011b; Ramasamy and Abbasi 2000) because it offers many advantages over wet anaerobic digestion. When compared to wet anaerobic digestion (WAD) systems, DAD requires less reactor volume, requires less energy input for heating and mixing, produces nutrient-rich fertilizer, and has a higher net energy yield (Cabral et al. 2014).

Agricultural wastes and livestock manure DAD (15–20 % TS) have recently been reported (Ahn et al. 2010; Di Maria et al. 2012; Kusch et al. 2008). In a suitability and economic feasibility assessment survey of on-farm DAD for solid manure, crop residues, spoiled hay and silage, and energy crops in Europe, Schäfer et al. (2006) concluded that although DAD is suitable for on-farm conditions, the ideal technologies have not been invented yet. Brown et al. (2012) found that volumetric productivity of DAD (18 % TS) is two to seven times greater compared to that of WAD (5 % TS) for lignocellulosic

Table 1 Nomenclature

ADF	Acid detergent fiber
ADL	Acid detergent lignin
CH ₄	Methane
CO ₂	Carbon dioxide
DAD	Dry anaerobic digestion
DSRDC	Dairy and Swine Research and Development Centre, Agriculture and Agri-Food Canada, Sherbrooke, Quebec
GC	Gas chromatograph
H ₂ S	Hydrogen sulfide
ISM	Inoculum-substrate mixture
ISR	VS-based inoculum-to-substrate ratio
NDF	Detergent fiber
N L	Normalized liter (at standard temperature and pressure, 273 K and 101.3 kPa)
OLR	Organic loading rate
PDAD	Psychrophilic dry anaerobic digestion
PDAD-SBR	Psychrophilic dry anaerobic digestion–sequence batch reactor
SBR	Sequence batch reactor
SMY	Specific methane yields
TCL	Treatment cycle length
TCOD	Total chemical oxygen demand
TS	Total solids
VFAs	Volatile fatty acids
VS	Volatile solids
WAD	Wet anaerobic digestion

substrates (wheat straw, switchgrass, corn stover, leaves, and waste paper) based on 30-day batch incubation. Most farm-based anaerobic digestion processes in northern Italy are operated at an organic loading rate (OLR) of 1–3 kg volatile solids (VS)m⁻³ and retention time greater than 40 days.

Recently, Agriculture and Agri-Food Canada has been developing a psychrophilic dry anaerobic digestion process in sequence batch reactor (PDAD-SBR) for dairy cow feces without and with different levels of straw bedding to stabilize cow manure and convert its biodegradable organics to methane. Massé and Saady (2015a) reported a long-term operation (252 days) of PDAD-SBR for digesting cow feces (TS 13–16 %) with an average specific methane yield (SMY) of 222±27.2 NL CH₄kg⁻¹ VS fed. Moreover, they demonstrated successful operation at TS of 27 % and OLR of 3 g total chemical oxygen demand (TCOD) kg⁻¹ inoculum day⁻¹ for 273 days with an average SMY of 182.9±16.9 NL CH₄kg⁻¹ VS fed (Massé and Saady 2015b). Increasing the OLR fed to a bioreactor is a basic engineering design objective to decrease the bioreactor volume (Luning et al. 2003) and reduce its construction costs. Nevertheless,

not many studies are published on the effect of OLR on the performance of dry anaerobic digestion. The goals of this study were to increase the OLR fed to PDAD-SBR of cow feces and wheat straw (27 % TS in feed) by 130–160 % and assess its feasibility at high OLRs (7.0 and 8.0 g TCOD kg⁻¹ inoculum day⁻¹) in long-term study (84 days). Wheat straw has been chosen because it is regularly used as bedding on Canadian dairy farms.

Materials and methods

Experimental setup

The experimental work consisted of two simultaneous experiments of operating psychrophilic (20 °C) anaerobic sequence batch reactors (SBRs) at two organic loading rates (OLRs) of 7.0 and 8.0 g TCOD kg⁻¹ inoculum day⁻¹ to digest a mixture of cow feces and wheat straw (TS=27 % in feed). The treatment cycle length (TCL) has been maintained at 21 days measured from the time of feeding to the next feeding. The effects of OLR on CH₄ production and process stability have been assessed during a long-term study (84 days) consisting four successive treatment cycles.

Bioreactor

Two sets of duplicate 40-L cylindrical barrel bioreactors have been operated as SBR at TCL of 21 days in a temperature-controlled room (20 °C). The working volume ranged between 7.2 and 8.5 L based on the volumes of the feed and inoculum used. The reactors were fitted with two gas lines—one for purging nitrogen gas immediately after feeding the substrate to maintain an anaerobic condition, and the second to release and quantify the biogas produced (Fig. 1).

Inoculum and substrate

The initial inoculum was obtained in a laboratory-scale (30 L; TS=27 %) psychrophilic (20 °C) anaerobic

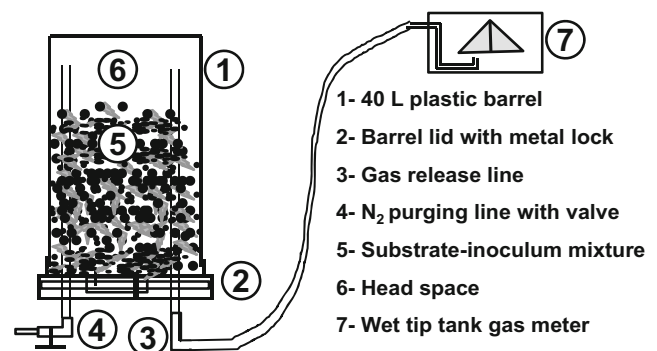


Fig. 1 Schematic diagram of the dry anaerobic digester

sequence batch reactor fed with fresh dairy manure and wheat straw (27 % TS); the performance of the seeding inoculum has been reported previously (Saady and Massé 2013). Starting from the second cycle forward, 6 kg of the digestate from the previous cycle has been used as inoculum for the next cycle in each reactor. Fresh feces from dairy cows were collected at the experimental farm of the Dairy and Swine Research and Development Centre, Agriculture and Agri-Food Canada, Sherbrooke, Quebec (DSRDC). Feces were collected on wood boards, before getting in contact with urine and bedding, transferred into a plastic drum, and stored at 4 °C (for a maximum of 30 days before feeding), and triplicate representative samples have been taken from each batch of cow feces fed to the reactors in each treatment cycle after thorough mixing and homogenization and before feeding the reactors. Physico-chemical characteristics of the inoculum, cow feces, and inoculum-substrate mixture (ISM) before feeding bioreactors are given in Table 2. Two different batches of cow feces from the same experimental farm at DSRDC, Sherbrooke, Quebec, were used during the four cycles as indicated in Table 2 (the first batch has been used for cycles 1 and 2, while the second batch has been used for cycles 3 and 4). Wheat straw was harvested at the DSRDC's experimental farm during fall 2011 and fall 2012 and chopped (3 mm) using a laboratory mill (Thomas Wiley Laboratory Mill Model 4, Arthur H. Thomas Company, Philadelphia, PA).

The substrate and inoculum have been mixed manually for 5 min during feeding. Every week, the content of the bioreactor is mixed manually for 5 min before sampling the content to ensure that a homogenous and representative sample is taken. No mixing took place during other time of the treatment cycle; therefore, the process can be considered as a static dry anaerobic digestion.

Organic loading rate

Wheat straw and cow feces were mixed manually to obtain the desired substrate TS content (27 %) while maintaining the design OLR of 7.0 and 8.0 g TCOD kg⁻¹ inoculum day⁻¹ (equivalent to 5.15±0.06 and 5.86±0.02 g VS kg⁻¹ inoculum day⁻¹). The mass of inoculum, feces, and/or straw fed to each bioreactor at the beginning of the successive cycles, the organic loading rate (OLR), and VS-based inoculum-to-substrate ratio (ISR) are given in Table 3.

The organic loading rate (OLR) has been calculated based on the masses of VS and TCOD of the substrate fed (Table 3). ISR was expressed in kilograms of total VS fed per kilogram VS of inoculum. The ISR was 1.66±0.02 at OLR of 7.0 g TCOD kg⁻¹ inoculum day⁻¹ and 1.45±0.02 at OLR of 8.0 g TCOD kg⁻¹ inoculum day⁻¹.

Biogas measurement

Biogas volume produced was measured daily using calibrated wet tip gas meters while the biogas components (CH₄, H₂S, CO₂) were determined weekly using a Hach Carle 400 AGC gas chromatograph (GC) (Chandler Engineering, Houston, TX) at 85 °C with a helium gas flow rate of 30 mL min⁻¹. The GC calibration was performed weekly with a standard gas (27.3 % CO₂, 1.01 % N₂, 71.16 % CH₄, 0.53 % H₂S). Methane production is reported in normalized liters (NL CH₄). Total cumulative CH₄ yield was established at the end of each digestion cycle. Specific CH₄ yield for a TCL of 21 days was calculated for each cycle as the ratio of CH₄ produced over the mass of VS fed to the reactor at the beginning of the cycle.

Analytical methods

Samples were collected from each bioreactor and analyzed weekly for volatile fatty acids (VFAs), TS, VS, and pH. Total chemical oxygen demand (TCOD) was determined before and after each treatment cycle. TCOD, TS, VS, alkalinity, and pH were determined using standard methods (APHA 1992). VFA concentration was measured with a PerkinElmer gas chromatograph model 8310 (PerkinElmer, Waltham, MA), equipped with a DB-FFAP high-resolution column.

Fiber analysis

The complex substrate (cow feces and wheat straw) was subjected to fiber analysis to determine their content of cellulose, hemicellulose, and lignin. Hemicellulose can be calculated as the difference between neutral detergent fiber (NDF) and acid detergent fiber (ADF), cellulose as the difference between acid detergent fiber and acid detergent lignin (ADL) (Bauer et al. 2009).

Results

Further to the characteristics of cow feces given in Table 2, it contained TCOD of 155.2 and 169.0 g kg⁻¹ during cycles 1–2 and 3–4, respectively. The fiber content of cow feces fed during cycles 1–2 was 24.39 % (cellulose), 19.28 % (hemicellulose), and 12.74 % (lignin) while that fed during cycles 3–4 contained 26.33 % (cellulose), 16.35 % (hemicellulose), and 10.67 % (lignin). Wheat straw had a TCOD of 1097 g kg⁻¹ while its fiber composition was 38.61 % (cellulose), 25.14 % (hemicellulose), and 7.3 % (lignin). Throughout the experiments, the concentration of H₂S was always less than 0.06 %.

Table 2 Physico-chemical characteristics of the inoculum and inoculum-substrate mixture at the beginning of each digestion cycle

OLR	Cycle	Substrate	pH	TS (%)	VS (%)	Acetate (g kg ⁻¹)	Propionate (g kg ⁻¹)	Butyrate (g kg ⁻¹)
7	1	Inoculum	8.4±0.0	20.0±0.1	17.7±0.1	0.70±0.05	0.22±0.10	0.13±0.06
		Cow feces	6.71	11.5±0.2	10.1±0.1	3.9±0.2	0.96±0.05	0.50±0.03
		ISM	8.2±0.1	22.5±0.5	20.1±0.3	1.64±0.19	0.42±0.04	0.25±0.09
	2	Inoculum	7.9±0.1	20.2±0.1	18.0±0.0	0.11±0.01	0.02±0.01	0.00±0.00
		Cow feces	6.71	11.5±0.2	10.1±0.1	3.9±0.2	0.96±0.05	0.50±0.03
		ISM	7.8±0.1	21.8±0.3	19.6±0.3	0.93±0.31	0.30±0.13	0.22±0.11
	3	Inoculum	8.0±0.0	20.2±0.6	18.0±0.4	0.14±0.01	0.05±0.01	0.05±0.01
		Cow feces	6.74	12.6±0.3	11.5±0.1	4.01±0.09	1.41±0.03	2.35±0.02
		ISM	8.0±0.3	21.7±0.3	19.5±0.3	0.79±0.49	0.21±0.15	0.05±0.04
	4	Inoculum	7.9±0.0	19.8±0.2	17±0.1.7	0.24±0.01	0.10±0.02	0.07±0.01
		Cow feces	6.74	12.6±0.3	11.5±0.1	4.01±0.09	1.41±0.03	2.35±0.02
		ISM	7.7±0.4	22.9±0.4	20.6±0.4	1.54±0.70	0.60±0.35	0.31±0.20
	1–4	Wheat straw	NA	89±1.2	84.9±0.5	NA	NA	NA
8	1	Inoculum	8.3±0.1	20.4±1.1	18.0±1.0	0.50±0.03	0.09±0.02	0.05±0.01
		Cow feces	6.71	11.5±0.2	10.1±0.1	3.9±0.2	0.96±0.05	0.50±0.03
		ISM	8.2±0.1	21.3±0.7	19.1±0.8	1.68±0.73	0.40±0.27	0.32±0.24
	2	Inoculum	8.0±0.1	20.2±0.5	18.0±0.5	0.12±0.01	0.02±0.01	0.06±0.0
		Cow feces	6.71	11.5±0.2	10.1±0.1	3.9±0.2	0.96±0.05	0.50±0.03
		ISM	7.8±0.2	22.3±0.7	20.3±0.7	1.89±0.45	0.50±0.09	0.02±0.21
	3	Inoculum	8.2±0.2	20.7±0.5	18.5±0.6	0.14±0.02	0.04±0.00	0.72±0.01
		Cow feces	6.74	12.6±0.3	11.5±0.1	4.01±0.09	1.41±0.03	2.35±0.02
		ISM	7.8±0.3	22.2±1.6	20.2±1.6	1.38±0.78	0.37±0.27	0.15±0.10
	4	Inoculum	7.9±0.1	20.4±0.4	18.3±0.5	0.19±0.02	0.05±0.02	0.03±0.02
		Cow feces	6.74	12.6±0.3	11.5±0.1	4.01±0.09	1.41±0.03	2.35±0.02
		ISM	7.7±0.0	22.0±1.2	19.8±1.0	1.19±0.32	0.45±0.11	0.15±0.16
	1–4	Wheat straw	NA	89±1.2	84.9±0.5	NA	NA	NA

ISM inoculum-substrate mixture immediately after feeding, NA not available, OLR organic loading rate, TS total solids, VS volatile solids

Methane production

The profiles of methane production expressed as specific methane yield (SMY) in the bioreactors are shown in Fig. 2. The maximum SMYs calculated during the successive cycles

are given in Table 4. The average of the SMY at OLR of 7.0 and 8.0 g TCOD kg⁻¹ inoculum day⁻¹ is 147.1±17.2 and 143.2±11.7 NL CH₄kg⁻¹ VS fed, respectively. The coefficient of variation for the duplicate reactors operated at OLR of 7.0 g TCOD kg⁻¹ inoculum day⁻¹ was 3.2±1.3 %, while at

Table 3 Details of the organic load fed to each bioreactor at the beginning of the successive cycles

	Cycle	Feces (kg)	Straw (kg)	ISR (VS-based)	Organic loading			
					TCOD fed (g)	VS fed (g)	g TCOD kg ⁻¹ inoculum day ⁻¹	g VS fed kg ⁻¹ inoculum day ⁻¹
In all cycles, the reactors have been inoculated with 6 kg of culture which has been transferred from the previous cycle. Feed TS has been kept at 27 % in all cycles TCOD total chemical oxygen demand, VS volatile solids	1	2.05	0.514	1.68	882.0	644.0	7.0	5.10
	2	2.05	0.514	1.68	882.0	644.0	7.0	5.10
	3	2.117	0.478	1.63	882.0	650.0	7.0	5.20
	4	2.117	0.478	1.63	882.0	650.0	7.0	5.20
	1	2.343	0.587	1.47	1008	736.0	8.0	5.84
	2	2.343	0.587	1.47	1008	736.0	8.0	5.84
	3	2.419	0.546	1.43	1008	742.0	8.0	5.88
	4	2.419	0.546	1.43	1008	742.0	8.0	5.88

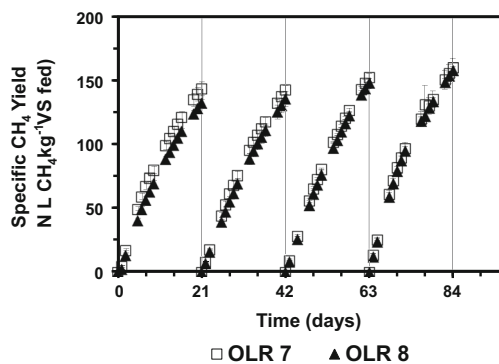


Fig. 2 Specific methane yield profiles for the psychrophilic dry anaerobic digestion of cow feces and wheat straw (27 % TS)

OLR 8.0 g TCOD kg⁻¹ inoculum day⁻¹, it was 2.8±1.1 %. The overall averages of the SMY at both OLR are not statistically different (based Tukey’s multiple comparison test at 95 % confidence interval).

The percentage of methane in biogas ranged between 53.3±0.9 and 56.4±0.3 with an overall average of 54.5±1.1 and 54.7±1.1 at OLR of 7.0 and 8.0 g TCOD kg⁻¹ inoculum day⁻¹, respectively (Fig. 3). No statistical difference in the percentage of CH₄ in biogas could be detected between inoculum fed at 7.0 and 8.0 g TCOD kg⁻¹ inoculum day⁻¹.

Notice that wheat straw provided 69.2±2.1 % of fibers in the feed at OLR of 7 and 8 g TCOD kg⁻¹ inoculum day⁻¹. Generally, wheat straw provided 61.7±2.6 % of the feed TCOD, 65.2±3.0 % of the feed VS, and 19.2±0.9 % of feed mass (Supplementary Table S1).

The overall average of the specific CH₄ production rate (NL CH₄kg⁻¹ VS day⁻¹) of the replicate bioreactors at OLR of 7.0 and 8.0 g TCOD kg⁻¹ inoculum day⁻¹ is 7.1±0.4 and 6.7±0.6 NL CH₄kg⁻¹ VS day⁻¹, respectively. The values of the overall averages of specific CH₄ production rate at OLR of 7.0 and 8.0 g TCOD kg⁻¹ inoculum day⁻¹ are not significantly different from each other (based on Tukey’s multiple comparison test at 95 % confidence interval).

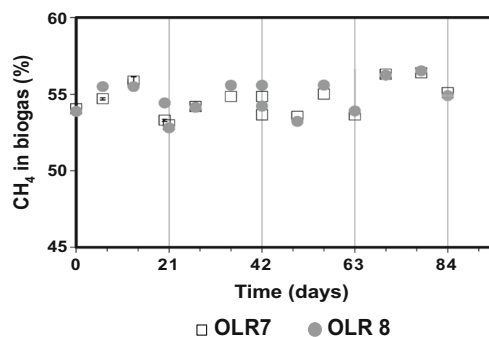


Fig. 3 Percentage of methane in biogas for the psychrophilic dry anaerobic digestion of cow feces and wheat straw (27 % TS)

Volatile fatty acid production

The profiles of VFAs produced in the replicate bioreactors were almost identical (data not shown). A typical VFA profile during a typical cycle operated at OLR of 7.0 g TCOD kg⁻¹ inoculum day⁻¹ is shown in Fig. 4. Throughout the successive cycles at OLR of 7.0 and 8.0 g TCOD kg⁻¹ inoculum day⁻¹, acetic acid concentration peaked immediately after feeding to levels between 1500 and 2000 mg L⁻¹ but was consumed within a week in all replicate bioreactors, and its concentrations were maintained within 100±50 mg L⁻¹ indicating that methanogenesis reaction from acetate was not a rate-limiting step. Similarly, propionic acid peaked to levels between 500 and 750 mg L⁻¹ after feedings and was consumed within a week to levels of 200 mg L⁻¹ and after another week to levels close to the detection limits of the instrument (50±10 mg L⁻¹). The profile of proinoic acid in the replicate bioreactots was similar to that of acetic acid.

Butyric acid peaked also to levels between 500 and 750 mg L⁻¹ after feedings and was consumed within a week to levels of 250 mg L⁻¹ and then to levels close to the detection limits of the instrument (25±10 mg L⁻¹) within another week. The concentrations of other volatile fatty acids (isobutyric, isovaleric, and valeric acid) were less than

Table 4 Specific methane yield and rate of methane production for the psychrophilic dry anaerobic digestion of cow feces and wheat straw (27 % TS)

Cycle number	OLR (g TCOD kg ⁻¹ inoculum day ⁻¹)	OLR (g VS fed kg ⁻¹ inoculum day ⁻¹)	SMY (NL CH ₄ kg ⁻¹ VS)	SMY per Kg TCOD (NL CH ₄ kg ⁻¹ TCOD)	Rate of CH ₄ production per total VS (NL CH ₄ kg ⁻¹ VS day ⁻¹)
1	7.0	5.10	143.3±5.5	104.6±4.0	6.8±0.6
2	7.0	5.10	142.5±3.3	104.0±2.4	6.8±0.5
3	7.0	5.2	152.2±3.2	112.1±2.3	7.2±0.4
4	7.0	5.2	159.9±7.6	117.8±5.5	7.6±0.8
1	8.0	5.88	131.5±3.2	104.3±14.9	6.3±0.4
2	8.0	5.88	135.3±6.0	91.9±18.6	6.4±0.7
3	8.0	5.88	146.4±5.7	108.8±12.8	7.0±0.6
4	8.0	5.88	156.8±5.6	116.0±12.5	7.5±0.6

The numbers given are averages and standard deviations of duplicate bioreactors. *TCL*=21 days for all cycles at both OLRs examined *OLR* organic loading rate, *TCOD* total chemical oxygen demand, *VS* volatile solids, *SMY* specific methane yield, *NL* normalized liters

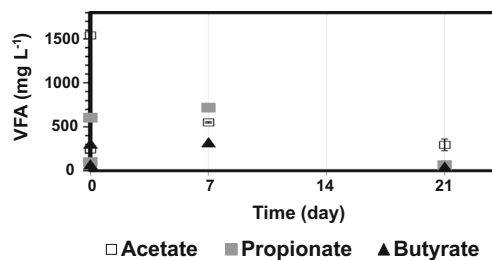


Fig. 4 Volatile fatty acid profile in a typical cycle of cow feces and wheat straw psychrophilic dry anaerobic digestion

100 mg L⁻¹ immediately after feeding and less than 50 mg L⁻¹ during the remaining time of the TCL. No differences have been observed among the VFA profiles associated with the different OLRs applied.

Solids reduction

The profiles of the TS and VS in the replicate bioreactors were similar (data not shown). A typical profile of VS during a typical cycle operated at OLR of 7 g TCOD kg⁻¹ inoculum day⁻¹ is shown in Fig. 5. Generally, the TS and VS contents of the reactor content at the end of the treatment cycles averaged around 20.9±0.3 and 18.7±0.1 % at OLR of 7.0 g TCOD kg⁻¹ inoculum day⁻¹, respectively, while at OLR of 8.0 g TCOD kg⁻¹ inoculum day⁻¹, they averaged around 21.1±0.4 and 19.0±0.2 %, respectively. A mass balance of the VS revealed that the VS removal efficiency averaged around 57.4±0.5 and 60.5±5.7 % at OLR of 7.0 and 8.0 g TCOD kg⁻¹ inoculum day⁻¹, respectively.

Discussion

Feasibility of PDAD-SBR has been demonstrated for digesting cow feces (13–16 % TS) during long-term operation (252 days) with an average specific methane yield (SMY) of 222±27.2 NL CH₄kg⁻¹ VS fed at OLR of 5.0 g TCOD kg⁻¹ inoculum day⁻¹ (Massé and Saady 2015a). Furthermore, the feasibility of digesting dairy manure (cow feces and wheat straw) at 27 % TS in PDAD-

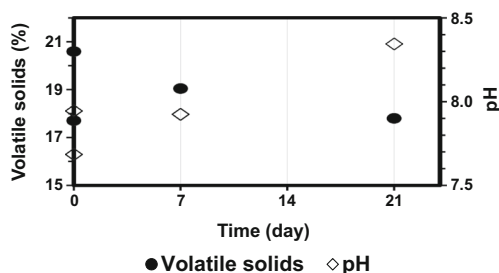


Fig. 5 pH and volatile solid profiles in a typical cycle of cow feces and wheat straw psychrophilic dry anaerobic digestion

SBR has successfully been demonstrated at OLR of 3 g TCOD kg⁻¹ inoculum day⁻¹ in long-term operation (273 days); it yielded an average SMY of 182.9±16.9 NL CH₄kg⁻¹ VS fed (Massé and Saady 2015b). The results of this study answers the following question: Is it possible to have a high-rate PDAD-SBR? In this study, the OLR has been increased by 130–165 % and the performance and stability of psychrophilic anaerobic sequence batch bioreactors in terms of specific methane yield, volatile fatty acid levels, and TS and VS removal have been assessed during four successive cycles (84 days) at OLR of 7.0 and 8.0 g TCOD kg⁻¹ inoculum day⁻¹.

Specific methane yield (overall average of 146.0±9.9 NL CH₄kg⁻¹ VS of cow feces and wheat straw) obtained during the eight successive cycles at both OLRs examined with a variation coefficient of 8.6 % indicates a stable and reproducible process. Based on Tukey's multiple comparison test at 95 % confidence interval, the overall averages of the SMY at each OLR are not statistically different which means that the performance of the psychrophilic dry anaerobic digestion in sequence batch reactor operated at TCL of 21 days has not been affected by increasing the OLR by 14.3 % (from 7.0 to 8.0 g TCOD kg⁻¹ inoculum day⁻¹). The ability of the PDAD-SBR process to withstand such increase in the OLR without showing signs of instability indicates its robustness.

The substrate mixture fed contained cow feces and wheat straw; both contain lignocellulose fibers such as cellulose which requires long retention time to be biodegraded by the anaerobic consortia of microorganisms, and its hydrolysis has been reported to be the rate-limiting step (Noike et al. 1985) particularly when it is fed in solid substrate or particulate form such as cattle manure (Myint and Nirmalakhandan 2006). Therefore, sequence batch reactor is suitable for digesting lignocellulosic substrates because it offers long solid retention time which allows wheat straw to contribute to methane production. In this study, the SBR at 21-day TCL with the ISR used provided sufficient solid retention time (SRT) to degrade lignocellulose fibers. Nevertheless, the TCL is relatively short when compared to previously published data (Table 5) for dry anaerobic digestion conducted under mesophilic conditions. The ISR used in this study might look relatively high compared to the values used in WAD; however, the VS-based ISR values used in this study (1.43–1.68) are justified by the hard conditions. Notice that at psychrophilic conditions, the rate of reaction is lower than that at mesophilic conditions. Moreover, the type of substrate and its TS content are much higher than those used in WAD.

Volatile fatty acid (acetate, propionate, butyrate, and other short-chain fatty acids) profiles indicated that acetogenesis and methanogenesis steps were not rate-limiting. The VFA concentrations and the progress of methane production during the successive cycles suggest a pseudo steady state condition and indicate that acetogenic and methanogenic reactions

Table 5 Comparative performance of dry anaerobic digestion of cow manure and wheat straw

Substrate	TS (%)	ISR	OLR (g VS kg ⁻¹ inoculum day ⁻¹)	Temp. (°C)	TCL (days)	SMY (L kg ⁻¹ VS)	Reference
Dairy manure and wheat straw	27	1.64±0.03 ^a	5.14±0.04	20	21	145.8±13.4 ^b	This study
	27	1.45±0.02 ^a	5.80±0.00	20	21	143.2±11.7 ^b	This study
Dairy manure and wheat straw	27	5.55±0.03 ^a	2.23±0.18	20	21	182.9±16.9 ^b	Massé and Saady (2015b)
Dairy manure	13-16	1.9 ^c	3.2	20	21	222.0±27.2 ^b	Massé and Saady (2015a)
	13-16	2.1 ^c	2.7	20	21	189.9±2.7 ^b	
	13-16	2.5 ^c	2.1	20	21	184.9±24 ^b	
Cattle manure		NR		35	80	112–191	Sunarso et al. (2012)
Sheep manure (DAD)		1.36 ^a	0.81	35	94	184 ^d	Blanco et al. (2010)
Animal manure		1.0 ^a	2.4	35	47	216±40	Frigon et al. (2012)
Cow manure and crop silage		NR	4.45			237.6	Comino et al. (2010)
			5.15			249 (stable)	
			7.78			61.6 (failure)	
Cattle slurry		NR	2.44	35	NR	400	Mahnert and Linke (2009)
			3.25			370	
Cow manure and crop silage		NR	4.25	39	75	360 ^d	Lindorfer et al. (2008)
Beef manure plus straw	18	NR	3.2	32	28	181	Schäfer et al. (2006)
Dairy manure, straw, and oat husk	17	NR	3.4	38	22	160	Schäfer et al. (2006)
			4.1	38	22	84	
Rice straw and corn stover inoculated with (1:1) sewage sludge, pig manure	25	0.2	NR	26–28	198	382	Sun et al. (1987)
	30				198	423	
	35					34	
Fresh horse manure and straw	20	0.2	NR	37	28	146	Kusch et al. (2008)
					42	175	
					72	208	
Cow manure and wastewater sludge (2:3 mass ratio)	16	0.2	0.35 ^e	35	63	328	Li et al. (2011a)
						251	
						319	
Beef manure and grass silage (85 %:15 %)	28	NR	0.9 ^f	35	100	227	Schafer et al. (2006)

TCL treatment cycle length, ISR inoculum-to-substrate ratio, TS total solids, VS volatile solids, SMY specific methane yield, DAD dry anaerobic digestion

^a VS-based

^b Normalized to standard pressure and temperature

^c Mass-based

^d SMY units are NL CH₄kg⁻¹ VS

^e OLR units are kg TCOD kg⁻¹ VS

^f OLR units are kg TCOD kg⁻¹ VS day⁻¹

proceeded fairly well. Hence, hydrolysis was the rate-limiting step. The relative stability of the pH profile around 7.8±0.6 (Fig. 5) was due to the high alkalinity (10.14±0.86 g CaCO₃ L⁻¹) of the reactor content which was contributed mainly by cow feces.

The VS removal efficiency achieved was on average 57.4±0.5 and 60.5±5.7 %, and the percentage of methane in biogas was on average 54.5±1.1 and 54.7±1.1 at OLR of 7.0 and 8.0 g TCOD kg⁻¹ inoculum day⁻¹, respectively. However, there is potential for more VS degradation and increase in

the specific methane yield by increasing the TCL. The latter would require larger bioreactor size.

The results have been compared with the reported performance of mesophilic and thermophilic DAD of various substrates (Table 5) because no relevant data are available in the literature on the performance of psychrophilic DAD. The overall average yield (145.1±2.8 NL CH₄kg⁻¹ VS of cow feces and wheat straw) of the eight cycles operated at OLR of 7.0 and 8.0 g TCOD kg⁻¹ inoculum day⁻¹ (equivalent to 5.15±

0.06 and 5.86 ± 0.02 g VS fed kg^{-1} inoculum day^{-1}) obtained in this study after 21 days of psychrophilic (20 °C) incubation is smaller than the yield 160 NL $\text{CH}_4\text{kg}^{-1}$ VS of dairy manure, straw, and oat husk (TS 17 % at OLR of 3.4 g VS kg^{-1} inoculum day^{-1}) reported by Schäfer et al. (2006) for Jarna biogas plant in Sweden which operates at 38 °C and retention time of 22 days. The data reported from Jarna plant is for steady state condition where the inoculum was adapted to the substrate and the operation condition for 3 years at the time of the study reported by Schäfer et al. (2006). The average SMYs (147.1 ± 17.2 and 143.2 ± 11.7 NL $\text{CH}_4\text{kg}^{-1}$ VS fed) obtained in this study at OLR of 5.15 ± 0.06 and 5.86 ± 0.02 g VS kg^{-1} inoculum day^{-1} are 20 % smaller than the 181 L $\text{CH}_4\text{kg}^{-1}$ VS of beef manure and straw (TS 18 % and OLR of 3.2 g VS kg^{-1} inoculum day^{-1}) at 32 °C and retention time of 28 days reported by Schäfer et al. (2006) (Table 5). Compared to Schäfer et al. (2006) result, the current study demonstrated an increase of 30 % in the feed TS, increase of 62.5 to 81.25 % in OLR, and a decrease in the treatment cycle length of 25 %. The increase of the OLR from 3.2 to 5.8 g VS kg^{-1} inoculum day^{-1} results in a 55 % reduction in the required volume of the bioreactor. Notice that the high yields (>250 NL $\text{CH}_4\text{kg}^{-1}$ VS fed) reported by Li et al. (2011a, b) was for TS of 16 % and TCL of 63 days in mesophilic anaerobic digestion of cow manure and wastewater sludge. Similarly, the yield reported by Sun et al. (1987) in Table 5 have been obtained for long retention times (168 days) and low OLR (0.35 g TCOD kg^{-1} inoculum day^{-1}) in mesophilic operations with substrate including corn stover. Achieving a stable dry anaerobic digestion of cow manure and wheat straw (TS of 27 %) at high rate (OLR of 7.0 and 8.0 g TCOD kg^{-1} inoculum day^{-1} equivalent to 5.1 ± 0.0 and 5.8 ± 0.0 g VS kg^{-1} inoculum day^{-1}) in psychrophilic conditions and over long-term operation (189 days) is a significant improvement given that 30 % TS has been recently identified as a threshold above which methanogenesis was strongly inhibited in mesophilic (35 °C) cardboard batch anaerobic digestion (Abbassi-Guendouz et al. 2012).

The specific methane yields obtained in this study provide evidence that at OLR of 7.0 and 8.0 g TCOD kg^{-1} inoculum day^{-1} , PDAD of cow manure and straw is technically feasible at TS 27 % and is as efficient as mesophilic DAD. Therefore, a high-rate psychrophilic (20 °C) dry anaerobic digestion of cow feces and wheat straw (feed TS=27 %) in sequence batch reactor with VS-based inoculum-to-substrate ratio of 1.66 ± 0.02 at OLR of 7.0 g TCOD kg^{-1} inoculum day^{-1} and 1.45 ± 0.02 at OLR of 8.0 g TCOD kg^{-1} inoculum day^{-1} is a feasible biotechnology for cold-climate regions.

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