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Feasibility and performance of high-rate psychrophilic dry anaerobic digestion of high solids content dairy manure

Noori M. Cata Saady[1] · Daniel I. Massé[1]

Abstract

Purposes The main purpose of the R&D was to develop a novel high-rate psychrophilic dry anaerobic digestion (PDAD) for processing a mixture of cow feces and wheat straw (CFWS) in sequencing batch bioreactor (SBR) operated at high organic loading rate (OLR). The main research question was to assess the feasibility of operating the PDAD-SBR at OLR of 9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1}.

Methods Two pairs of 40 L duplicate bioreactors located in a temperature-controlled room (20 °C) were operated as sequencing batch reactors (SBRs) and were fed CFWS (27 % total solids) at OLRs of 9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1} during three successive treatment cycles of 21 days each. The biogas production, biogas composition, volatile fatty acids, and volatile solids were monitored to assess the performance of the PDAD-SBRs.

Results The PDAD process fed with cow feces and wheat straw (TS of 27 %) at OLR 9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1} (6.53 ± 0.06 and 7.27 ± 0.12 g VS kg^{-1} inoculum day^{-1}) resulted in average specific methane yields (SMYs) of 134.0 ± 7.7, 136.4 ± 4.9 $_N$L CH_4 kg^{-1} VS fed, respectively, with an average volatile solids removal of 35 % at the ends of treatment cycles (21 days).

Conclusions This study reports for the first time successful operation of psychrophilic (20 °C) dry anaerobic digestion (PDAD) of cow feces with wheat straw (CFWS) at 27 % total solids and OLR of 9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1} in relatively short treatment cycle length (21 days).

Introduction

Stabilization of biodegradable organics in solid manure from livestock industry has been a challenge because conventional wet anaerobic digestion (WAD; Table 1) requires dilute influent which could be pumped in and out of the digester, mixed, and heated, thus digesters of large volume would be required. So far, conventional anaerobic digestion (AD) of cattle manure has been ineffective because of increased energy input for mixing and heating. In addition, the large volumes of digesters decrease the specific methane yield (SMY) per unit volume of digester. Theoretically, increasing the biodegradable organics content of the substrate increases the specific volumetric methane yield. Solid cattle manure (total solids = 25 %; Demirer and Chen 2008) is richer in its biodegradable organics than dilute slurries (TS = 5 %). Therefore, developing a static dry anaerobic digestion (DAD; TS > 15 %) to replace WAD would increase the specific volumetric methane yield and eliminate energy expenditure for mixing. Based on 30 days batch anaerobic digestion of lignocellulosic substrates, Brown et al. (2012) found that volumetric productivity of DAD (18 % TS) is 2- to 7-times greater than that of WAD (5 % TS). Moreover, if psychrophilic rather than mesophilic or thermophilic operation is adopted less energy input for heating would be required and the net energy recovery would be increased. Schäfer et al. (2006) stated that DAD is

✉ Daniel I. Massé
Daniel.masse@agr.gc.ca

[1] Dairy and Swine Research and Development Centre, Agriculture and Agri-Food Canada, Sherbrooke, QC J1M 0C8, Canada

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Table 1 Nomenclature

AD	Anaerobic digestion
ADF	Acid detergent fiber
ADL	Acid detergent lignin
APHA	American Public Health Association
CFWS	Cow feces and wheat straw mixture
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_2S	Hydrogen sulfide
HRT	Hydraulic retention time
ISM	Inoculum-substrate mixture
ISR	VS-based inoculum-to-substrate ratio
N_2	Nitrogen gas
NDF	Detergent fiber
$_NL$	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
PSBR	Psychrophilic sequence batch reactor
SBR	Sequence batch reactor
SMY	Specific methane yields
STP	Standard temperature and pressure (273 K; 1 atm)
TCL	Treatment cycle length
TCOD	Total chemical oxygen demand
TS	Total solids
VFAs	Volatile fatty acids
VS	Volatile solids
WAD	Wet anaerobic digestion

suitable for on-farm conditions, but there is need to invent and introduce ideal technologies in terms of substrate handling, loading and unloading of bioreactors. However, some technical advancement has been achieved since then. Currently, DAD is relatively a new biotechnology for agricultural wastes and livestock manure (15–20 % TS). Recently, several studies investigated DAD of undiluted cow manure without bedding in mesophilic and thermophilic batch reactor (Ahn et al. 2010; Böske et al. 2014, 2015; Di Maria et al. 2012; Kusch et al. 2008; Li et al. 2014). Moreover, a novel process for psychrophilic (20 °C) DAD of undiluted cow manure without bedding (TS = 12–16 %) in a sequence batch reactor has been reported recently (Massé and Saady 2015b) and for cow feces with bedding (wheat straw; TS = 27 %; Massé and Saady 2015c). These researchers reported a SMY of 182.9 ± 16.9 $_NL$ CH_4 kg^{-1}

VS fed at organic loading rate (OLR) of 3.0 g TCOD kg^{-1} inoculum day^{-1} in sequence batch reactor (SBR). Investors prefer increasing the OLR to decrease the bioreactor size and lower the capital and operation costs. The effect of OLR on the performance of cow manure DAD has not been studied sufficiently. Hence, feasibility of high-rate PDAD of cow feces with bedding (TS = 27 %) is still unanswered question. The 27 % TS in feed was targeted because cattle in sheds with straw bed produce manure of around 27 % TS (Demirer and Chen 2008). This study aims to:

1. Increase the OLR by 12.5 and 25 % (to 9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1}) in comparison with previously published data (8.0 g TCOD kg^{-1} inoculum day^{-1}).
2. Demonstrate the feasibility of PDAD of CFWS (TS = 27 %) at the above-stated conditions.
3. Evaluate effect of increasing substrate OLR and decreasing the inoculum-to-substrate ratio (ISR) on the long-term performance.

Materials and methods

Experimental setup

The experiments were designed to assess the influence of the OLR (9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1}) on CH_4 production and process stability of PDAD. The feed was CFWS at feed total solids of 27 % over three successive cycles (63 days) at each OLR. The reactors operated at both OLRs were operated in parallel to exclude the variation in feed and other experimental conditions.

Bioreactor

The experimental work utilized cylindrical plastic drums (volume = 40 L) as bioreactors. The bioreactors were operated as psychrophilic sequential batch reactors (PSBR) at 21 days treatment cycle length (TCL). The cycle is composed of the following steps: day 1: inoculating the reactor with mixed anaerobic culture obtained from the previous cycle, feeding the substrate, and mixing the inoculum and substrate (all were in aerobic condition); days 1–21: anaerobic reaction; and day 21: emptying the digestate (in aerobic condition) and starting the next cycle as in step 1. The bioreactors were kept in a temperature-controlled room (20 °C). Two gas lines were connected to the reactor's cover (Fig. 1). The first gas line was used for flushing nitrogen (N_2) gas immediately after feeding phase to create the anaerobic atmosphere inside the bioreactor. The second gas line was used as gas outlet connected to a

Table 2 Physicochemical characteristics of the cow feces fed in each treatment cycle

Parameter	Cow feces		Wheat straw
	Cycle 1	Cycles 2–3	Cycles 1–3
pH	6.74	7.02	NA
Total chemical oxygen demand (TCOD; g kg^{-1})	169.0	180.0	1097
Total solids (TS; %)	12.6	12.8	89.0
Volatile solids (VS; %)	11.5	11.8	84.9
Volatile fatty acids (g kg^{-1})			
Acetate	4.01	2.93	NA
Propionate	1.41	0.86	NA
Butyrate	2.35	0.55	NA
Fiber components (% of dry matter)			
Cellulose (% of dry matter)	26.33	24.69	38.61
Hemi-cellulose (% of dry matter)	16.35	26.06	25.14
Lignin (% of dry matter)	10.67	10.77	7.3

Values given are averages of duplicate samples

NA not applicable

Table 3 Details of organic loading of psychrophilic dry anaerobic digestion in sequencing batch reactor

Parameters	Cycle 1	Cycle 2	Cycle 3	Cycle 1	Cycle 2	Cycle 3
OLR (g TCOD kg^{-1} inoculum day^{-1})	9.0	9.0	9.0	10.0	10.0	10.0
OLR (g VS fed kg^{-1} inoculum day^{-1})	6.6	6.5	6.5	7.4	7.2	7.2
Cow feces (kg)	2.721	2.685	2.685	3.024	2.983	2.983
Wheat straw (kg)	0.614	0.593	0.593	0.683	0.659	0.659
ISR (VS-based)	1.28	1.26	1.48	1.25	1.20	1.37
Feed TCOD/VS ratio	1.36	1.38	1.38	1.36	1.38	1.38
TCOD fed (g)	1134	1134	1134	1260	1260	1260
VS fed (g)	835	821	821	928	912	912

In all cycles the reactors have been inoculated with 6 kg of inoculum (culture) which has been transferred from the previous cycle. The feed TS has been kept at 27 % in all cycles

OLR organic loading rate, TCOD total chemical oxygen demand, VS volatile solids, ISR inoculum-to-substrate ratio

wet tip tank meter which measures the volume of the biogas generated. Details of the wet tip tank have been given elsewhere (Saady and Massé 2015).

Inoculum and substrate

The initial inoculum (used in the first cycle) was mixed anaerobic culture (TS = 27 %) obtained from a laboratory scale PDAD-SBR digesting dairy cow feces with bedding (wheat straw; 27 % TS; Massé and Saady 2015c). Dairy cow feces was obtained from the dairy farm of the Dairy and Swine Research and Development Centre (DSRDC), Sherbrooke, Quebec. Feces were collected into plastic drums and measures were taken to avoid contamination of the collected feces with urine and bedding. The feces drums were stored at 4 °C until the feces has been fed to the reactors. Table 2 gives the physicochemical characteristics of the cow feces fed in the successive treatment cycles.

Wheat straw has been chopped to 3 mm particles using a laboratory mill (Thomas Wiley Laboratory Mill Model 4, Arthur H. Thomas Company, Philadelphia, PA, USA). Chopped wheat straw and cow feces were mixed and homogenized using manual scoop to achieve a substrate TS content (27 %) and OLRs of 9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1} (6.53 ± 0.06 and 7.27 ± 0.12 g VS kg^{-1} inoculum day^{-1}).

Organic loading rate

Table 3 gives the mass of inoculum, feces, and/or straw fed to each bioreactor at the beginning of the successive cycles. The OLR has been calculated using the masses of VS and TCOD of the CFWS fed (Tables 2, 3). Table 4 gives the physicochemical characteristics of the inoculum and inoculum-substrate mixture (ISM) prior to being fed to the bioreactors. The inoculum-to-substrate ratio (ISR) was expressed in kg VS of inoculum per kg of total VS fed and OLR was expressed in g of

Table 4 Physico-chemical characteristics of the inoculum and inoculum-substrate mixture (ISM) at the beginning of each digestion cycle

Parameters	Cycles 1, 2, 3	Cycles 1, 2, 3
OLR (g TCOD kg^{-1} inoculum day^{-1})	9.0	10.0
Alkalinity ISM	9.25	9.8
pH Inoculum	8.16	8.05
pH ISM	7.75	7.76
TS Inoculum (%)	19.67	20.5
TS ISM (%)	22.6	21.9
VS Inoculum (%)	18.3	18.6
VS ISM (%)	20.8	19.9
Acetate Inoculum (g kg^{-1})	0.27	0.30
Acetate ISM (g kg^{-1})	1.07	1.28
Propionate Inoculum (g kg^{-1})	0.06	0.15
Propionate ISM (g kg^{-1})	0.32	0.33
Butyrate inoculum (g kg^{-1})	0.03	0.67
Butyrate inoculum (g kg^{-1})	0.18	0.15

Values given are averages of six samples

OLR organic loading rate, *ISM* inoculum-substrate mixture, *TS* total solids, *VS* volatile solids

TCOD fed per kg of inoculum, and in g of total VS fed per kg of inoculum per day. The average of ISR was 1.34 ± 0.12 at OLR of 9.0 g TCOD kg^{-1} inoculum day^{-1} and 1.27 ± 0.09 at OLR of 10.0 g TCOD kg^{-1} inoculum day^{-1}.

Sampling

Samples of the inoculum and substrate mixture (ISM) were taken on day (0) immediately after feeding and on day (21) at the end of the treatment cycle. It was also sampled on day (7) by opening the reactor, mixing and homogenizing its content manually for 5 min to obtain a representative sample. The sample obtained on day (21) represented the digestate (effluent) of which 6 kg were used as inoculum in the next cycle. Anaerobic condition was established on day (0) immediately after feeding by flushing the reactor with nitrogen. The biogas was sampled by 10 mL plastic syringe through septa-sealed gas sampling port installed on the second gas line.

Biogas measurement

Daily measurements were taken for the volume of the biogas produced using wet tip gas meters. The components of biogas (CH_4, H_2S, CO_2) were determined weekly on a MicroGC 490 gas chromatograph (GC) (Agilent Technologies, China).

Specific CH_4 yield (SMY) in each cycle was determined by dividing the cumulative CH_4 volume produced (within 21 days) by the mass of volatile solids (VS) fed to the bioreactor at the start of the cycle. Methane (CH_4) production is given in normalized litres ($_N$L CH_4 by correcting its volume to standard temperature and pressure (STP; 273°K; 1 atm).

Analytical methods

Total solids (TS), volatile solids (VS), and pH were determined weekly while total chemical oxygen demand (TCOD) and alkalinity were measured at the beginning and end of the treatment cycle according to Standard Methods (APHA 1992).

The levels of volatile fatty acids (VFAs) from acetic to isovaleric acids were measured weekly on Perkin Elmer GC model 8310 (Perkin Elmer, Waltham, MA, USA) equipped with an autosampler to facilitate the analysis. The column, GC setup and analytical protocol have been reported elsewhere (Massé et al. 2003).

Fiber analysis

The cellulose, hemicellulose, and lignin components of the fiber have been determined through fiber analysis performed on duplicate samples of the raw cow feces and wheat straw separately for each batch of these substrates. Hemicellulose has been determined by subtracting the quantity of acid detergent fiber (ADF) from the quantity of neutral detergent fiber (NDF). Cellulose was determined by subtracting the quantity of acid detergent lignin (ADL) from the quantity of acid detergent fiber (ADF) (Bauer et al. 2009).

Statistical methods

Data presented in tables and shown in figures are the means and standard deviations of duplicate measurements. Statistical analysis was carried out using Minitab v. 14

statistical software (Minitab Inc., PA, USA). Data were analyzed by one-way ANOVA, followed by post hoc multiple comparisons (Tukey's test) with a confidence level of 95 % (i.e., $p < 0.05$).

Results and discussion

Methane production

The long-term performance of PDAD-SBR was assessed in three cycles at each OLR (9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1}). Figure 2 shows the cumulative specific methane yield (SMY) obtained in each treatment cycle while Table 5 gives the maximum SMYs calculated during each treatment cycle.

The overall average SMY at OLR of 9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1} is 134.0 ± 7.7, 136.4 ± 4.9 $_N$L CH$_4$ kg^{-1} VS fed (97.6 ± 6.3 and
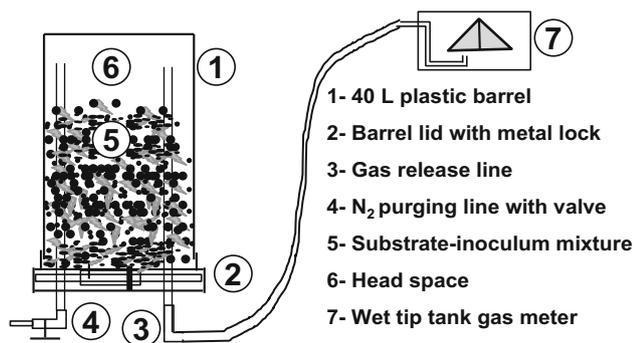
Fig. 1 Schematic diagram of the dry anaerobic digester (Massé and Saady 2015c)

1- 40 L plastic barrel
2- Barrel lid with metal lock
3- Gas release line
4- N$_2$ purging line with valve
5- Substrate-inoculum mixture
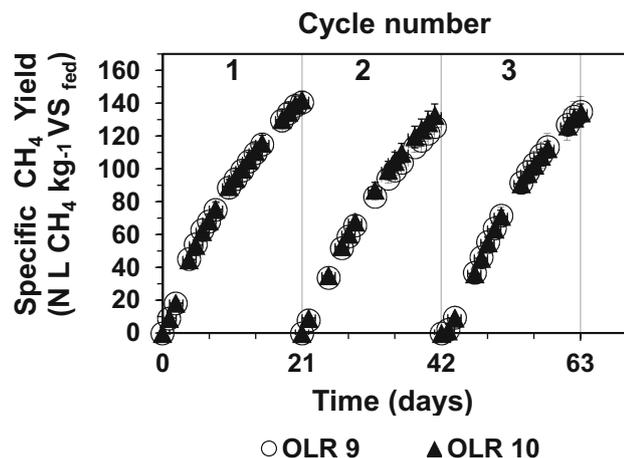6- Head space
7- Wet tip tank gas meter

Fig. 2 Specific methane yield for the psychrophilic dry anaerobic digestion of cow feces and wheat straw (27 % TS) at OLR = 9 (*open circle*) and OLR = 10 (*filled triangle*)

99.3 ± 4.5 $_N$L CH$_4$ kg^{-1} TCOD fed), respectively. The coefficient of variation of the SMY between the duplicate reactors fed at OLR 9.0 g TCOD kg^{-1} inoculum day^{-1} was 5.76 ± 2.80 % while for the reactors fed at OLR 10.0 g TCOD kg^{-1} inoculum day^{-1} it was 3.58 ± 0.92 % without a significant statistical difference according to Tukey's multiple comparison test at 95 % confidence interval. This indicates that the performance of the PDAD-SBRs was not influenced by the 11.1 % increase in OLR. It seems that the quality of cow feces fed affected the performance of psychrophilic anaerobic sequence batch reactor digesting 27 % total solids more than the increase in organic loading rate. The cow feces which has been fed during cycle 1 had lower TCOD and VS values ($\approx$169.0 g TCOD kg^{-1} feces, 11.1 % VS, respectively) in comparison with the feces fed in cycles 2–3 (180.0 g TCOD kg^{-1} feces, 11.2 % VS, respectively). However, the SMYs (140.4 ± 4.5 and 141.8 ± 5.4 $_N$L CH$_4$ kg^{-1} VS fed at OLR 9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1}, respectively) obtained during cycle 1 were greater than that obtained during cycle 2–3 (125.4 ± 3.3 and 132.4 ± 7.8 $_N$L CH$_4$ kg^{-1} VS fed at OLR 9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1} during cycle 2, respectively). This could be partially explained by the higher concentrations of VFAs in the feces used during cycle 1 compared to that fed in cycle 2–3. Cow feces fed in cycle 1 contained 37, 64.0, and 327 % more acetic, propionic, and butyric acids compared to the cow feces used in cycle 2–3.

Methane formed between 53.0 ± 0.6 and 57.6 ± 0.0 % of the biogas produced by volume. The overall average of methane percentage in biogas was 55.8 ± 1.4 and 55.6 ± 1.1 at OLR of 9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1}, respectively (Fig. 3) and they were statistically the same. Somayaji and Khanna (1994) reported that CH$_4$ percentage in biogas was around 58–59 % for wet anaerobic digestion of 10 % TS feed (cow manure and wheat straw mixture) at hydraulic retention time (HRT) of 40 days. They observed that changing the percentage of wheat straw total solids in feed from 20 to 40, 60, and 80 % did not affect the percentage of methane in biogas though it affected the SMY. On the contrary, Hashimoto (1983) reported 31 % CH$_4$ in biogas when 7.8 % TS feed of 50:50 (mass ratio) of cow manure and wheat straw was anaerobically digested at HRT of 15 days. Increasing the mass ratio of wheat straw in feed to 75 and 90 % decreased CH$_4$ proportion in biogas to 16 %.

Wheat straw contributed on average between 67.4 ± 0.4 % of the feed total fibers; 58.1 ± 1.1 % of the feed TCOD; 61.8 ± 0.6 % of the feed VS and 18.2 ± 0.2 of feed mass at the examined OLRs (Table S1). Notice that

Table 5 Specific methane yields and rates of methane production for the psychrophilic dry anaerobic digestion of cow feces and wheat straw (27 % TS)

Parameters	Cycle 1	Cycle 2	Cycle 3	Cycle 1	Cycle 2	Cycle 3
Organic loading rate (OLR)						
g TCOD kg^{-1} inoculum day^{-1}	9.0	9.0	9.0	10.0	10.0	10.0
g VS fed kg^{-1} inoculum day^{-1}	6.6	6.5	6.5	7.4	7.2	7.2
Cow feces TCOD (g kg^{-1})	169.0	180.0	180.0	169.0	180.0	180.0
Specific methane yield (SMY)						
$_NL$ CH_4 kg^{-1} VS	140.4 ± 4.5	125.4 ± 3.3	136.2 ± 10.5	141.8 ± 5.4	132.4 ± 7.2	134.9 ± 5.1
$_NL$ CH_4 kg^{-1} TCOD	103.4 ± 3.3	90.8 ± 2.4	98.6 ± 7.6	104.4 ± 4.0	95.8 ± 5.2	97.7 ± 3.7
Rate of CH_4 production ($_NL$ CH_4 kg^{-1} VS day^{-1})	6.7 ± 0.2	6.0 ± 0.2	6.5 ± 0.5	6.8 ± 0.3	6.3 ± 0.4	6.4 ± 0.3

The numbers given are averages and standard deviations of duplicate bioreactors

The treatment cycle length (TCL) in each cycle was 21 days

Values given are averages of duplicate samples

TCOD total chemical oxygen demand, *VS* volatile solids, *CH₄* methane
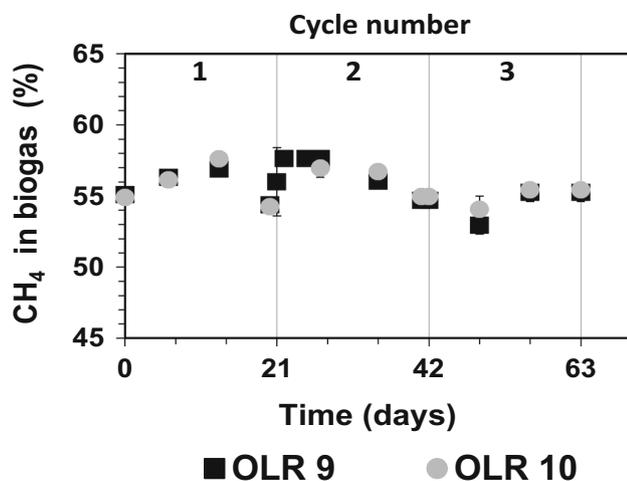

Fig. 3 Percentage of methane in biogas for the psychrophilic dry anaerobic digestion of cow feces and wheat straw (27 % TS)

SMY calculated at OLR 9.0 g TCOD kg^{-1} inoculum day^{-1} during cycle 1 was greater than that obtained in cycle 2 and 3 by 10.7 and 3.0 %, respectively. Similarly, SMY obtained at OLR 10.0 g TCOD kg^{-1} inoculum day^{-1} during cycle 1 was greater than that obtained in cycle 2 and 3 by 6.6 and 4.9 %, respectively. The lower SMY obtained during cycles 2–3 compared to that obtained in cycle 1 (at both OLRs examined) could be partially due to the less wheat straw fiber fed. Notice that the mass of wheat straw fiber in the feed for cycle 2 and 3 were 13 and 15 g less than that fed in cycle 1, respectively; Table S1. Lignocellulose fiber such as cellulose biodegrades slowly during anaerobic digestion thus stipulates long retention time. Hydrolysis reactions of lignocellulose fiber are rate limiting because of physicochemical and compositional factors in the structure of fiber (Kumar et al. 2009; Noike

et al. 1985). This phenomenon is especially important when the substrate is in solid or particulate form similar to cow manures (Myint and Nirmalakhandan 2006).

Somayaji and Khanna (1994) reported that changing the proportion of wheat straw in the feed mixture affected the SMY but not CH_4 percentage in biogas while Hashimoto (1983) reported that it affected both of them.

Overall average specific CH_4 production rate ($_NL$ CH_4 kg^{-1} VS day^{-1}) obtained at OLR of 9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1} is 6.4 ± 0.4 and 6.5 ± 0.2 $_NL$ CH_4 kg^{-1} VS fed, respectively. These values are statistically the same which implies that there is no difference in terms of biogas production rate between the operation at OLR of 9.0 or 10.0 g TCOD kg^{-1} inoculum day^{-1}.

Except the recently published studies (Massé and Saady 2015a, c; Saady and Massé 2015), no information has been published on the psychrophilic anaerobic digestion of CFWS at high total solids (>20 %) and high OLR (>6 g VS kg^{-1} inoculum day^{-1}). Therefore, Table 6 has been compiled by comparing the results with the recent data published by the authors of this study. Moreover, the results were compared with the data from mesophilic and thermophilic DAD of relevant substrates. Generally, the SMY ($_NL$ CH_4 kg^{-1} VS fed) obtained from digesting CFWS mixture (27 % TS) at 20 °C during 21 days decreased from 182.9 ± 16.9 to 136.4 ± 4.9 as the OLR increased from 3.0 to 10.0 g TCOD kg^{-1} inoculum day^{-1} (2.2 ± 0.2 to 7.3 ± 0.1 g VS kg^{-1} day^{-1}). The overall average SMYs of the 3 cycles fed at OLR of 9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1} (6.53 ± 0.06 and 7.27 ± 0.12 g VS fed kg^{-1} inoculum day^{-1}) obtained in this study after 21 days of psychrophilic (20 °C) incubation were 134.0 ± 7.7 and 136.4 ± 4.9 $_NL$ CH_4 kg^{-1} VS fed, respectively. These SMYs are lower than the 160 $_NL$ CH_4 kg^{-1} VS of dairy manure, straw, and oat husk (fed at

Table 6 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)	References
Dairy manure and wheat straw	27	1.45 ± 0.02	7.3 ± 0.1	20	21	136.4 ± 4.9	This study
	27	1.64 ± 0.03	6.3 ± 0.1	20	21	134.0 ± 7.7	This study
	27	1.66 ± 0.01	5.2 ± 0.1	20	21	143.2 ± 11.7	Massé and Saady (2015a)
	27	1.45 ± 0.02	5.8 ± 0.0	20	21	147.1 ± 17.2	Massé and Saady (2015a)
	27	1.4 ± 0.05	4.4 ± 0.1	20	21	175.4 ± 11.9	Saady and Massé (2015)
	27	1.7 ± 0.05	3.7 ± 0.1	20	21	193.8 ± 11.8	Saady and Massé (2015)
	27	2.9 ± 0.1	2.9 ± 0.1	20	21	179.8 ± 20.4	Saady and Massé (2015)
	27	5.56 ± 0.01	2.2 ± 0.2	20	21	182.9 ± 16.9	Massé and Saady (2015b)
Cattle manure	12	0.014–0.057		35	80	112–191	Sunarso et al. (2012)
Sheep manure (dry anaerobic digestion)		1.36 (mass based)	0.81	35	94	184[c]	Blanco et al. (2010)
Animal manure		1.0 (VS-based)	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[c]	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[a]	35	63	328	Li et al. (2011)
						251	
						319	
Beef manure and grass silage (85 %:15 %)	28	NR	0.9[b]	35	100	227	Schäfer et al. (2006)

TCL treatment cycle length, *VS* volatile solids, *NR* not reported

[a] Organic loading rate (OLR) units are kg TCOD kg^{-1} VS

[b] OLR units are kg TCOD kg^{-1} VS day^{-1}

[c] Specific methane yield (SMY) units are $_NL$ CH$_4$ kg^{-1} VS

TS 17 % and OLR of 3.4 g VS kg^{-1} inoculum day^{-1}) and digested at 38 °C during 22 days in Järna biogas plant in Sweden (Schäfer et al. 2006).

Compared to the data reported by Schäfer et al. (2006) for the Järna biogas plant, this study increased the OLR from 3.4 to 7.27 ± 0.12 g VS fed kg^{-1} inoculum day^{-1}, increased the substrate TS from 17 to 27 %, and decreased the operation temperature from 38 to 20 °C without losing more than 15.6 % of the SMY. Obviously, the increase in OLR, TS, and the decrease in operation temperature means

reduction in capital and operational costs and increase in the net energy yield and in the return-on-investment.

The average SMYs (134.0 ± 7.7 and 136.4 ± 4.9 $_N$-L CH$_4$ kg^{-1} VS fed) obtained in this study at OLR of 6.5 ± 0.1 and 7.3 ± 0.1 g VS kg^{-1} inoculum day^{-1} constitute 75 % of the SMY (181 L CH$_4$ kg^{-1} VS) obtained from digesting beef manure and straw mixture (TS 18 %) at OLR of 3.2 g VS kg^{-1} inoculum day^{-1} in mesophilic condition (32 °C) during 28 days (Schäfer et al. 2006; Table 6). In comparison with Schäfer's et al. (2006) result,

this study increased the feed TS by 30 %, increased the OLR by 103–128 %, and shortened TCL by 25 %. Increasing the OLR from 3.2 to 7.3 g VS kg^{-1} inoculum day^{-1} decreased the required bioreactor's size by 55 %. It is worthwhile to notice that digesting cow manure and wastewater sludge mixture (61 % TS) anaerobically at very low OLR (0.35 g TCOD kg^{-1} inoculum day^{-1}) and in relatively long TCL (63 days) yielded >250 $_N$L CH$_4$ - kg^{-1} VS fed (Li et al. 2011). Obviously, the yields of 382 $_N$L CH$_4$ kg^{-1} VS fed obtained by Sun et al. (1987) in Table 6 is not practical because of the excessive TCL (198 days) at mesophilic condition (26–28 °C) and that 50 % of the substrate was an energy crop (corn stover). Demonstrating a stable PDAD of cow manure and wheat straw (TS of 27 %) at high-rate OLR of 9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1} (6.3 ± 0.1   and 7.3 ± 0.1 g VS kg^{-1} inoculum day^{-1})   during   63 days comprising three successive cycles is an important and promising development. The feed TS of 27 % is very close to the threshold of 30 % above which methanogens were significantly inhibited at 35 °C during batch anaerobic digestion of cardboard (Abbassi-Guendouz et al. 2012).

Somayaji and Khanna (1994) reported that for 10 % TS feed of cow manure and wheat straw mixture a maximum SMY of 107 L kg^{-1} VS fed was obtained within 40 days HRT when the wheat straw contributed 40 % of the feed TS. The SMY decreased when wheat straw contribution to the feed TS increased to more than 40 % or decreased below it. A 100 L CH$_4$ kg^{-1} VS fed has been obtained by Hashimoto (1983) from digesting 7.8 % TS feed (50:50 cow manure and wheat straw) at 35 °C and 15 days HRT. The SMY decreased to 70 and 30 L CH$_4$ kg^{-1} VS fed when the wheat straw was increased to 75 and 90 % by mass in the feed.

The SMYs achieved in the present study prove that PDAD of cow manure and straw is possible at TS 27 % and OLRs of 9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1} and its efficiency is comparable to that of mesophilic DAD in terms of SMY and stability.

Effects of inoculum-to-substrate ratio (ISR)

The ISR examined in this study was decreased from 1.34 ± 0.12 at OLR of 9.0 g TCOD kg^{-1} inoculum day^{-1} to 1.27 ± 0.09 at OLR of 10.0 g TCOD kg^{-1} inoculum day^{-1} [corresponding to a percentage of substrate-to-inoculum (VS-based) of 74.6 and 78.7, respectively].

Generally, the conclusions reported in literature on the effect of ISR on SMY are variable. For example, decreasing the ISR from 3.33 to 1.67 caused 20 % decrease in the SMY from Okra beans refuse (9.5 % TS) mesophilic batch DAD (19 days incubation time; Zhou et al. 2011). Similarly, a decrease in VS-based ISR from 3.0 to 1.0

caused 16 % reduction in SMY during 20 days of batch mesophilic AD of maize (10 % TS; Raposo et al. 2006). On the contrary, for dry grind corn ethanol plant stillage decreasing the VS-based ISR from 3.67 to 1.83, 0.92 and 0.46, respectively, increased the SMY by 1.25, 10, and 14.2 %, respectively (Eskicioglu and Ghorbani 2011). These researchers found that the rates of VS and COD removal were lower between ISR 0.46 and 0.92 compared to those at ISR of 1.83 and 3.67. However, lower ISR led to higher percentages of CH$_4$ in biogas which resulted in larger SMYs. Obtaining 134.0 ± 7.7 and 136.4 ± 4.9 $_N$-L CH$_4$ kg^{-1} VS fed at ISR of 1.34 ± 0.12 and 1.27 ± 0.09 and TCL of 21 days at psychrophilic conditions (20 °C) is an important advancement in the development of psychrophilic dry anaerobic digestion compared to the SMY values reported by other studies as given in Table 6.

Volatile fatty acids (VFAs) production

The levels and variations of the VFAs measured in the replicate bioreactors were similar (data not shown). Table 4 gives the concentrations of major VFAs (acetate, propionate, and butyrate) at the beginning (ISM) and end (inoculum) of the treatment cycles conducted at OLR of 9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1}. Generally, at both OLRs examined and during all treatment cycles, acetic acid levels rose upon feeding to around 1500 ± 710 mg L^{-1} but were decreased within a week due to the consumption by methanogens. Acetic acid levels were kept around 500 ± 180 mg L^{-1} throughout the rest of the cycle revealing that conversion of acetate to methane was not a rate limiting reaction. Similarly, propionic acid levels rose to around 270 ± 110 mg L^{-1} upon feedings and were decreased to levels within the instruments' detection limit (30 ± 10 mg L^{-1}). Profiles of propionic and acetic acids in the replicate bioreactors were similar (data not shown). Except for acetate, generally slight increase in the VFAs concentrations has been observed in samples taken at day 7 which was due to the fast hydrolysis of readily biodegradable part of organics in cow feces.

Butyric acid rose also to concentrations around 170 ± 40 mg L^{-1} upon feedings and was decreased to around the instruments' detection limit (25 ± 10 mg L^{-1}). Isobutyric-, iso-valeric-, and valeric-acids levels were less than 100 mg L^{-1} after feeding and were kept under 50 mg L^{-1} during the rest of the TCL. Despite the difference in the OLRs applied the VFAs profiles were similar. Acetogensis reactions from propionate, butyrate and other short-chain fatty acids were not rate limiting as it can be inferred from these acids profiles.
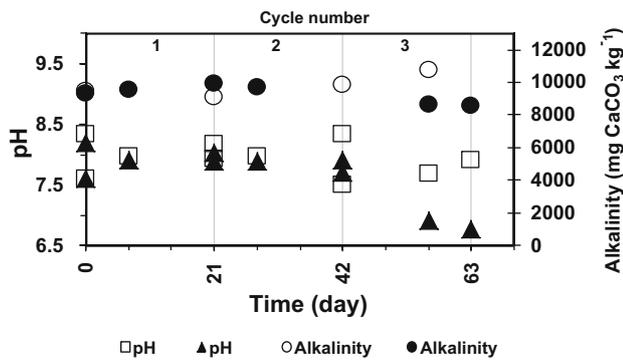
Fig. 4 Alkalinity and pH during psychrophilic dry anaerobic digestion (Open and closed legend are for OLR 9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1}, respectively. Two data points are shown at day zero, 21, and 42 for each parameter; one represents the inoculum and the other represents the inoculum-substrate mixture)

The VFAs concentrations and the profiles of CH$_4$ generation during the entire experiment revealed that acetogenesis and methanogenesis progressed sufficiently well. The pH profiles varied within a narrow range (from 7.76 ± 0.24 to 8.18 ± 0.12; Fig. 4) likely because of the high alkalinity levels (9270 ± 250 mg CaCO$_3$ kg^{-1}) of the digestion matrix.

Solids reduction

Total solids (TS) and volatile solids (VS) profiles in the duplicate bioreactors operated at each OLR were similar (data not shown). The VS removal averaged around 35.8 ± 4.3 and 34.4 ± 1.2 % at OLR 9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1}, respectively. Achieved VS removal is comparable to that (32 %) obtained by Somayaji and Khanna (1994) for wet anaerobic digestion of 10 % TS feed [cow manure and wheat straw (40:60 TS-based ratio)] at HRT of 40 days.

Conclusions

This study reports for the first time successful operation of psychrophilic (20 °C) dry anaerobic digestion (PDAD) of cow feces with wheat straw (CFWS) at 27 % total solids and OLR of 9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1} (6.53 ± 0.06 and 7.27 ± 0.12 g VS kg^{-1} inoculum day^{-1}, respectively) in relatively short treatment cycle length (21 days). The results demonstrated the feasibility of psychrophilic dry anaerobic digestion of cow feces and wheat straw in laboratory scale sequence batch reactor (SBR) with inoculum-to-substrate ratio (VS-based) between 1.34 ± 0.12 and 1.27 ± 0.09 at the examined OLRs. The average specific methane yields (SMY) were

134.0 ± 7.7 and 136.4 ± 4.9 $_N$L CH$_4$ kg^{-1} VS fed at OLR of 9.0 and 10.0 g TCOD kg^{-1} inoculum day^{-1}, respectively. Volatile solids removal at ends of the treatment cycles averaged around 35 %. Volatile fatty acids concentrations measured during the operation indicated that hydrolysis, but not acetogenesis or methanogenesis, was rate limiting. High-rate PDAD of animal manure and bedding (27 % TS) is possible, promising and has a strong potential for application in cold zones.

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