

HIGH-SOLID ANAEROBIC CO-DIGESTION OF FOOD WASTE AND DAIRY MANURE: A PILOT SCALE STUDY AT LOW-TO-MODERATE TEMPERATURE CONDITIONS

Rajinikanth Rajagopal^{1,*}, Bernard Goyette¹ and Jean-François Hince²

¹ Sherbrooke Research and Development Center, Agriculture and Agri-Food Canada, 2000 College Street, Sherbrooke, Québec J1M 0C8, Canada

² Bio-Terre Systems, 740 rue Galt Ouest, Sherbrooke, Québec J1H 1Z3, Canada

Article Info:

Received:
30 November 2018
Revised:
22 February 2019
Accepted:
22 February 2019
Available online:
31 March 2019

Keywords:

High-solid anaerobic digestion
Biomass adaptation
Fruit and vegetable waste
Low-to-moderate temperature
Methane yield

ABSTRACT

Treating organic solid wastes economically is a challenge, predominantly in cold and high-altitude regions. Objective of this research was to determine the operating strategies to reduce the start-up phase of high-solid anaerobic digestion (HSAD) process and to improve the digestion of food waste (mainly fruits and vegetable wastes [FWW]) with or without animal manure in a low-cost AD system at 20-25°C. In addition, this study aimed to obtain the basic design criteria for starting up of scaled-up HSAD system using adapted liquid inoculum. Inoculum to feedstock ratio was varied from 6:1 to 3:1. The organic loading rate (OLR) expressed as volatile solids (VS) and operational cycle length was varied from 0.44 -2.1 Kg_{VS} Kg_{inoculum}⁻¹ d⁻¹ and 33 -14d, respectively. Obtained results show that methane (CH₄) production from FWW was feasible at low-to-moderate temperature and specific methane yield of 0.4-0.6 L g_{VS}⁻¹ was observed even at high OLR. CH₄ conversion rates and its quality were not affected, while maintaining the operational stability (e.g. no acidification or VFA accumulations). CH₄ content reached over 60% and remained almost steady. Results also suggest that HSAD process at 25°C is comparatively efficient in saving heat energy and at the same time obtains the CH₄ values close to mesophilic conditions. This means that the smaller size digester (in the case of HSAD) is preferred as there is no waste dilution involved and also suitable for cold countries. Using this concept, livestock producers can play a role in reducing GHG emissions while also earning C-offset credits.

1. INTRODUCTION

Mining bioenergy from biomasses is an effective alternative energy resource that can be used in an environmental friendly way and requires less energy production (Zheng et al., 2012). Various biomasses derived from the carbonaceous wastes of human, livestock animals and natural resources that could be utilized as renewable energy resources. According to Environment Canada (2013), there has been growing interest in managing the organic-fraction of the municipal waste stream in recent years. In Canada, biodegradable material such as food waste (FW) represents nearly 40% of the residential waste stream; therefore diversion of organic materials is crucial to attain high diversion targets. Municipal wastes (table, activated sludge, etc.) are rich in protein, fat and fiber materials, which can be effectively treated using anaerobic digestion (AD) biotechnology. However, the high levels of non-fiber carbohydrate and fat contents present in FW could lead to fast acidification. Furthermore, accumulation of ammonia is attributed to its

high content of proteins (Braguglia et al., 2018). Co-digestion of FW organics with other organic fractions in AD can enhance a better nutritional supply and lessen the inhibiting elements, such as ammonia and fat/lipid (Khairuddin et al., 2015).

Currently, municipalities must pay to transport and dispose of these by-products in landfills/composting. An interesting option for municipalities would be to pay local farmers to receive and process these materials in AD bioreactors. For farmers, the co-digestion of cow manure (CM)+FW could increase the recovery of green energy, production of litter for the herd and organic nitrogen fertilizer for crops. Nevertheless, handling litter poses a significant cost on dairy operations. Largely, animal waste is considered an appropriate co-substrate due to its high alkalinity, low C/N ratio and diverse macro- and micronutrients required by the anaerobic consortium. From previous studies, it is worth to note that, the mixtures of FW and animal waste are typically composed by low percentages



of FW (Zhang et al. 2012; Agyeman and Tao, 2014) and thus, further research is necessary to process high amount of FW with minimal biological inhibition. Most of the AD treatment of FW is carried out predominantly at mesophilic temperatures (35-37°C) and few installations have been reported on the thermophilic AD (50-55°C) operation. In fact, thermophilic temperatures results in larger grade of digester imbalance and higher risk for ammonia inhibition than mesophilic conditions (Yang et al., 2015). Considering the cold weather conditions, mesophilic/thermophilic processes are constrained by the amount of energy needed to heat the AD systems to maintain the desired temperature. In addition, these processes are inhibited by free ammonia toxicity while treating N-rich wastes. Lowering the temperature to 20°C could assure good methane yields and stability by co-digesting FW+CM (Rajagopal et al., 2017); however limited information have been reported on the successful AD operation at temperatures below 35°C.

After the extensive research work done by Agriculture and Agri-Food Canada (AAFC) and Bio-Terre Systems (BTS) on the development of low-temperature AD system for treating high solid content wastes like solid separated animal manure and carcass (Massé et al., 2014, Rajagopal et al., 2014; Saady and Massé, 2015), focus is now put on the capacity of this new technological approach to play a capital role in the organic waste management challenges that several smaller municipalities are facing. Previous studies were performed using laboratory-scale digesters (30-120 L) to test different solid content manures with or without liquid inoculum percolation-recirculation mode of operation. It has been established that high solid AD can be successfully operates with manure up to 35% TS content (Saady and Massé, 2015). Thus, this paper aims to demonstrate the operational feasibility of high-solid anaerobic digestion (HSAD) system treating CM+FW at low-to-moderate temperature conditions and to encourage small-scale municipalities or farmers to adopt this technology at low cost. In addition digestion of FW as a sole feeding source with recirculation of liquid inoculum were performed. The special emphasis was given to evaluate the biodegradation of the organic waste and the optimal operation conditions (such as OLR, cycle length) based on the organic matter reduction and methane production. To obtain this, different approaches were used such that lab-scale operations (30 L active volume) were performed in parallel and compared with a scaled-up HSAD process (3 m³ active volume, that is to say 100 times bigger than the lab-scale digesters) to determine its feasibility of digesting high solid content fruit and vegetable wastes (FVW) with or without solid dairy manure. Liquid inoculum was used to start the pilot-scale operation and the biomass adaptation procedures (liquid to solid inoculum) were experimented.

2. METHODS AND MATERIALS

2.1 Experimental set-up and operating strategy

Scale-up testing was conducted in a container-type pilot digester (2.43 m wide x 2.43 m height x 6.1 m length) developed by Bio-Terre Systems and was installed outdoor. This system is equipped with insulation, heating system,

gas collection and liquid inoculum percolation-recirculation provisions. Provision was made to collect the liquid percolate using a 50.8 mm liquid collection valve located at the bottom of the container. The mixture of organic waste and solid inoculum were filled in four numbers of plastic bin-containers, each with a total capacity of 1 m³, primarily to ease the waste handling procedures compared to bulk loading of waste materials into the container itself. This pilot-scale container type digester can accommodate a total of 8 plastic bin-containers (i.e. total capacity of 8 m³). However, during this start-up phase of the study, four bin-containers were evenly filled with the mixture of solid inoculum and organic waste before being put in the digester container, such that a total volume of about 3 m³ of waste mixture were fed per cycle (33-14 d). The facility was also equipped with a weighing scale to measure the mass of all materials fed into the digester. Solid inoculum, organic waste, structural agent and the final mixture were weighted for mass balance purposes. The plastic bins handling were done with a tractor, while the inoculum and organic wastes were mixed using a S70 Bobcat. The mixed material was transferred to the respective bin-containers, weighted and loaded into the pilot-scale digester. Bioreactor was then sealed during the entire treatment cycle length. Biogas production, temperature and pressure were monitored through on-line. Sampling of the material was done at the beginning and the end of each cycle of operation.

At the start of each cycle, liquid inoculum was added to enhance the microbial activity. The liquid leachate was collected from the container with a 101.6 mm valve and was transferred into a storage bin (1 m³) by gravity. Once or twice a week, the leachate was recirculated back to the feed mixture to maintain good humidity level and to improve the waste-biomass contact. Throughout the treatment cycle, temperature in the container bioreactor was monitored daily using thermocouples installed in the container and also in the organic waste mixture. Hot summer fluctuated the temperatures in the head space up to 28-29°C and the heating system was adjusted to maintain the temperatures between 20-25°C accordingly.

In parallel, a portion of the feed mixture was collected separately and used to fill lab-scale digesters for closer monitoring purposes. 50-L HSAD digesters (30-L active volume) were operated in parallel at 25°C and gas production was monitored using mass flow meters. The operating protocol was maintained similar to that of pilot-scale experiments.

2.2 Inoculum and feedstock sources

The liquid inoculum was obtained from an on-going semi-industrial scale bioreactor treating diluted liquid CM and FW mixture at our research facility. 500-L of the liquid inoculum was taken to develop the solid inoculum, which was then mixed with 200 kg of straw bedding and 44 kg of raw solid dairy manure (without bedding, TS:19-20%). Fresh dairy CM was collected at the experimental farm of the Sherbrooke Research and Development Center. Feed mixture was evenly distributed into 4 bin-containers (3-m³ active volume) and was placed in the container type bioreactor for a period of 56 d primarily to allow a complete ad-

aptation of the inoculum. A second cycle was started with the addition of solid dairy manure and straw. Afterwards, for another 106 d of reaction, co-digestion was started by the addition of a small portion of FVW. Followed to this adaptation phase, 100% of the organic loading was provided by the FVW mixture.

Raw FVW was collected from local providers, which was then weighted and grinded with a rototiller mounted on a brush cutter. It mainly comprised potato and carrots peeling, salad, potato, apple, banana, pineapple, orange, broccoli, onion, carrots and other rotten food materials. General description and specific weight of the waste was taken at every waste collection point. Overall, the proportion of fruits was slightly higher than the vegetables (51%/49%). The material was stored at 4°C before utilisation.

2.3 Sampling and analysis

For a feeding operation, a batch of about 250 kg of waste was grinded together and evenly distributed into 150-L barrels to have a more homogenous feedstock (for e.g. 6 barrels received 40 kg each of the same grinded batch). Sampling was done accordingly by taking 10% of the distributed waste into the sampling box (for e.g. 40 kg distributed per barrel; 4 kg sample). Since majority of the material was comprised of rotten food, the grinding was quite easy and the particulate size was maintained smaller than 25.4 mm. For the inoculum and digested material, the sampling was done in the mixing container (steel garbage bin).

Liquid leachate samples (100-150 mL) were taken on a weekly basis from the liquid inoculum reservoir; and the HSAD reactors' samples were also collected at the beginning and at the end of each treatment cycle. These samples were analysed for total solids (TS), volatile solids (VS), volatile fatty acids (VFA), chemical oxygen demand (COD), pH and total Kjeldahl nitrogen (TKN). COD was determined by the closed reflux colorimetric method (APHA, 1992). The concentration of VFAs were measured using a Perkin Elmer gas chromatograph model 8310 (Perkin Elmer, Waltham, MA), mounted with a DB-FFAP high resolution

column. TS and VS were determined using standard methods (APHA, 1992). pH value was measured using PH meter (model, TIM840, France). TKN was analyzed using a Kjeltex auto-analyzer (TECATOR 1030, Tecator AB, Hoganas, Sweden) using the macro-Kjeldahl method (APHA, 1992). Daily biogas production was measured by GFM mass flow meters (Aalborg, USA). Biogas composition (methane, carbon dioxide, H₂S and nitrogen) was determined with a HachCarle 400 AGCgas chromatograph (Hach, Loveland, CO). The column and thermal conductivity detector were operated at 80°C.

3. RESULTS AND DISCUSSIONS

3.1 Start-up of pilot-scale HSAD and adaptation of solid biomass (Phase 1)

The initial two cycles were performed predominantly to adapt the liquid inoculum to the high solid content operation. Biogas quantity and compositions were followed to measure the biological activity. Figure 1 presents the specific methane yield (SMY) obtained for the initial two cycles of operation. At the end of the first cycle (37 d), the dairy manure was not entirely converted into biogas. For instance, the SMY reached only 58% of the expected conversion in comparison to laboratory scale operation (0.170 L_{CH₄} g_{VS}⁻¹). This indicates that residual organic material was still present in the digester and hence, second cycle was given a longer reaction period (i.e. about 127 d). At the end of second cycle, SMY obtained was about 0.244 L_{CH₄} g_{VS}⁻¹. But more importantly, the cumulative SMY for both the cycles was 0.166 L_{CH₄} g_{VS}⁻¹, which was the expected for the dairy manure digestion. For both cycles, the biogas composition was stabilized with a CH₄ content of around 40%.

3.2 Performance of pilot-scale HSAD treating FVW waste (Phase 2: Low-loading conditions)

From day 128 onwards, pilot HSAD system was fed with FVW and the performance was monitored in terms of organic matter destruction, VFA accumulation, biogas con-

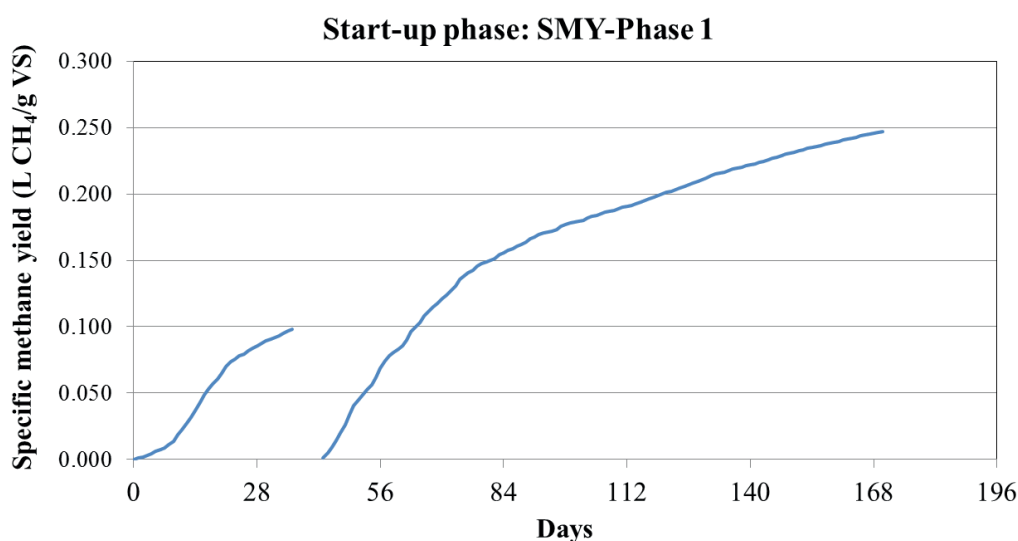


FIGURE 1: Start-up of HSAD and solid inoculum adaptation: Evolution of SMY.

centration and its quality, and SMY. For this phase of study, the ratio of solid inoculum to FW was maintained at 6:1 to limit the possibility of shock loading conditions to the bacteria. Organic loading rate (OLR) was maintained around $0.44\text{--}0.49 \text{ kg}_{\text{VS}} \text{ kg}_{\text{inoculum}}^{-1} \text{ d}^{-1}$ and the results in terms of biogas and cumulative methane production, and SMY are presented in Figure 2 (a-b). For both cycles, biogas production preceded fairly quick start-up with no lag phase after each feeding. It is to be noted that, about 77% of total biogas production was attained within 18 d and 12 d for cycle 3 (cycle length: 34 d) and 4 (cycle length: 28 d), respectively. High SMY values recorded for cycle 3 (i.e. $1.104 \text{ L}_{\text{CH}_4} \text{ g}_{\text{VS}}^{-1}$) in comparison to cycle 4 ($0.625 \text{ L}_{\text{CH}_4} \text{ g}_{\text{VS}}^{-1}$) were probably due to the digestion of residual VS accumulated from previous cycles of operation. Thus, longer reaction period was given to cycle 3 to allow a complete digestion of the remaining VS in the bioreactor. In addition, the biogas quality measured, especially after the feeding regime presented inconsistency, because of the operation procedure. As the feeding was done in a batch mode, the bioreactor was opened at the end of each cycle in order to be loaded with a new

material. As a result, the digester's headspace was filled with ambient air, which diluted the biogas for the initial few days of a cycle. It took five days for both cycles to ramp up the biogas quality to 55% of CH_4 in the measured biogas. At the end of treatment cycles 3 and 4, the methane concentration was 64% and 67%, respectively. The biogas with higher methane concentration indicates the good adaptation of biomass to the high-solid content process.

Organic matter mass balance was performed using TS and VS analysis. Inoculum, feedstock and digested material samples were taken and analysed to perform a mass balance approach. Table 1 illustrates the values from low loading operating conditions. The operation of the HSAD technology led to a great conversion of organic material into biogas based on the mass balance calculations. From the total 83 kg of VS fed, only 11 kg was accumulated in the inoculum at the end of the last cycle. It represented a reduction of 88%, which was the expected level of degradation for that nature of waste (>85%). The mass balance was not otherwise conclusive for the TS accumulations. Based on the values presented in Table 1, an accumula-

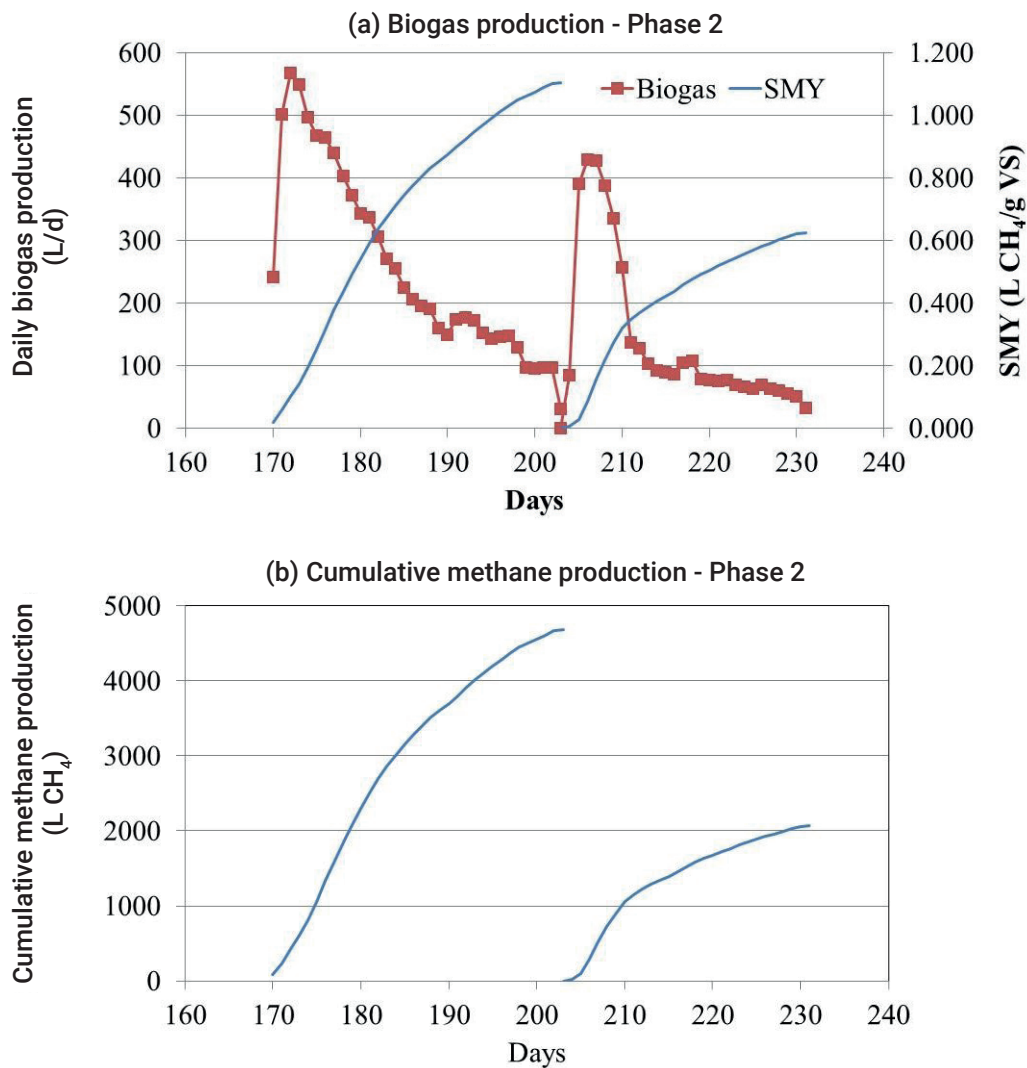


FIGURE 2 (a,b): Performance of Phase-2 (low OLR) HSAD process: a) Biogas production and SMY evolution; b) Cumulative methane production.

TABLE 1: Mass balance values for the low loading conditions (Inoculum to feed ratio of 6:1).

Cycle #	Inoculum start weight	Feedstock addition	TS fed	VS fed	Inoculum end weight	Inoculum TS in	Inoculum TS out	Inoculum VS in	Inoculum VS out
3	2 096 kg	389 kg	60 kg	45 kg	2 190 kg	306 kg	362 kg	236 kg	257 kg
4	1 673 kg	275 kg	26 kg	22 kg	1 673 kg	276 kg	285 kg	196 kg	188 kg
5	1 171 kg*	186 kg	18 kg	16 kg	1 225 kg	183 kg	184 kg	123 kg	120 kg
Total	4 940 kg	500 kg	104 kg	83 kg	5 088 kg	765 kg	831 kg	555 kg	565 kg

* Cycle 5 was done with 2/3 of the available inoculum. 1/3 remaining was used for the high loading testing

tion of 65 kg was present at the end of the treatment cycle. However, only 10 kg was provided from the VS accumulation and about 22 kg was resulted from the inorganic fraction of the solids. It is not apparent that all the residual 32 kg was contributed by the liquid inoculum (at 1.5 to 2% TS). Consequently, it was observed that rocks and sands accumulated in the inoculum. Those inert materials were probably introduced into the system during the bobcat operation and were not from the feeding substrates. It can be explained by some portion of the solid accumulation values measured in the mass balance.

The total organic mass increased to about 27% of the total feed weight. The majority of the weight loss was not due to the VS degradation but as a result of water content released throughout the digestion process. With about 90% water content, the feedstock ended-up with large quantity of water, which was then released while digesting the organic material. From the mass balance, 765 kg of water was included in the feedstock, in which, 548 kg was taken out of the inoculum in 3 cycles (liquid inoculum production and water vapor in the biogas). About 164 kg of water was accumulated in the solid inoculum increase. According to the data, about 53 kg of water (6.9% of all water) was not accounted from the balance. The three digestion cycles showed that high solid digestion of FW was effective in converting organic material into good quantity and quality of biogas. The mass balance indicates that for each kg of FW fed to the solid inoculum, about 0.27 kg was accumulated in the inoculum and about 0.65 kg of liquid inoculum was released.

3.3 Performance of pilot-scale HSAD treating FW waste (Phase 3: High-loading conditions)

The purpose of this phase of study was to increase the OLR by decreasing the ratio of inoculum to feedstock, such that same size bioreactor can process more waste materials with short retention times. From day 232 onwards, the proportion of solid inoculum to dairy manure was retained at 3:1. The similar operating strategy was followed for this phase of study as that of Phase 2. The same testing approach was used to follow this series of tests by using container bioreactor for the mass balance and the laboratory scale bioreactor for the biogas measurements. OLR was maintained around 1.6 to 2.1 $\text{kg}_{\text{VS}} \text{kg}_{\text{inoculum}}^{-1} \text{d}^{-1}$ and cycle length was controlled at 14-16 d. Results in terms of biogas and cumulative methane production, and SMY are presented in Figure 3 (a-b). Although, 5th and 6th cycles were operated at high OLR conditions, AD preceded fairly quick start-up with no lag phase after feeding with FVW. More than

75% of the total biogas production was attained within 7-d for both cycles. SMY values in the range of 0.400-0.520 $\text{L}_{\text{CH}_4} \text{g}_{\text{VS}}^{-1}$ were recorded. The obtained values are comparable to the AD of semi-dry mixed municipal FW (SMY: $0.401 \pm 0.01 \text{L}_{\text{CH}_4} \text{g}_{\text{VS}}^{-1}$) [Rajagopal et al., 2017]

The short retention times and high OLR conditions seemed to have a little impact on the inoculum for the 5th cycle of operation (SMY: $0.400 \text{L}_{\text{CH}_4} \text{g}_{\text{VS}}^{-1}$) but not enough to imbalance the process. However, for the subsequent cycles (Figure 3, a-b), the SMY increased by 30%, which indicates the good activity of the biomass. This was confirmed by the less VFA accumulations (total content below 900 mg L⁻¹) and high buffering capacity of the digester (digester pH: 7.2-7.5). pH of the substrate was acidic (4.0-4.5), but however, in the reactor it was in the neutral range. There was no sign of inhibition or nutrient deficiency at these operating conditions.

The methane concentration during the first 4 days of operation was around 58% (for both cycles). At the end of the treatment cycle, the same was increased to 66% and 63%, respectively. Better performance were obtained even at higher loading conditions and short treatment cycles due to the good adaptation of the biomass. These results were comparable to that of laboratory scale study, particularly in terms of SMY (i.e. $0.4\text{-}0.5 \text{m}^3 \text{CH}_4 \text{kg}_{\text{VS}}^{-1}$), and methane content of about 62-72%. The quality of biogas in the pilot-scale digester increased with time and remained almost stable thereafter. This specifies that the smaller size digester (in the case of HSAD) is preferred as there is no waste dilution involved. This could reduce a major part in the capital investment on the construction of digesters and also suitable for cold countries. Further experiments were continued to optimise the inoculum to feed ratio, high OLRs and short treatment cycle lengths.

Since the container type bioreactor was not providing accurate biogas production, operation follow up was done using mass balance approach. All the inoculum, feedstock and digested materials were weighted in the container before and after the digestion process. Samples were taken and analysed to perform a mass balance analysis. Values from all high loading cycles are presented in Table 2. Mass balance based on organic matter values was performed using TS and VS analysis. Inoculum, feedstock and digested material samples were taken and analysed to perform a mass balance approach. Table 2 presents the values from high-loading operating conditions. The increase of the loading rate modified the mass balance obtained from the last two cycles. While organic fractions were converted up to 87% in the first three cycles, the high loading operation

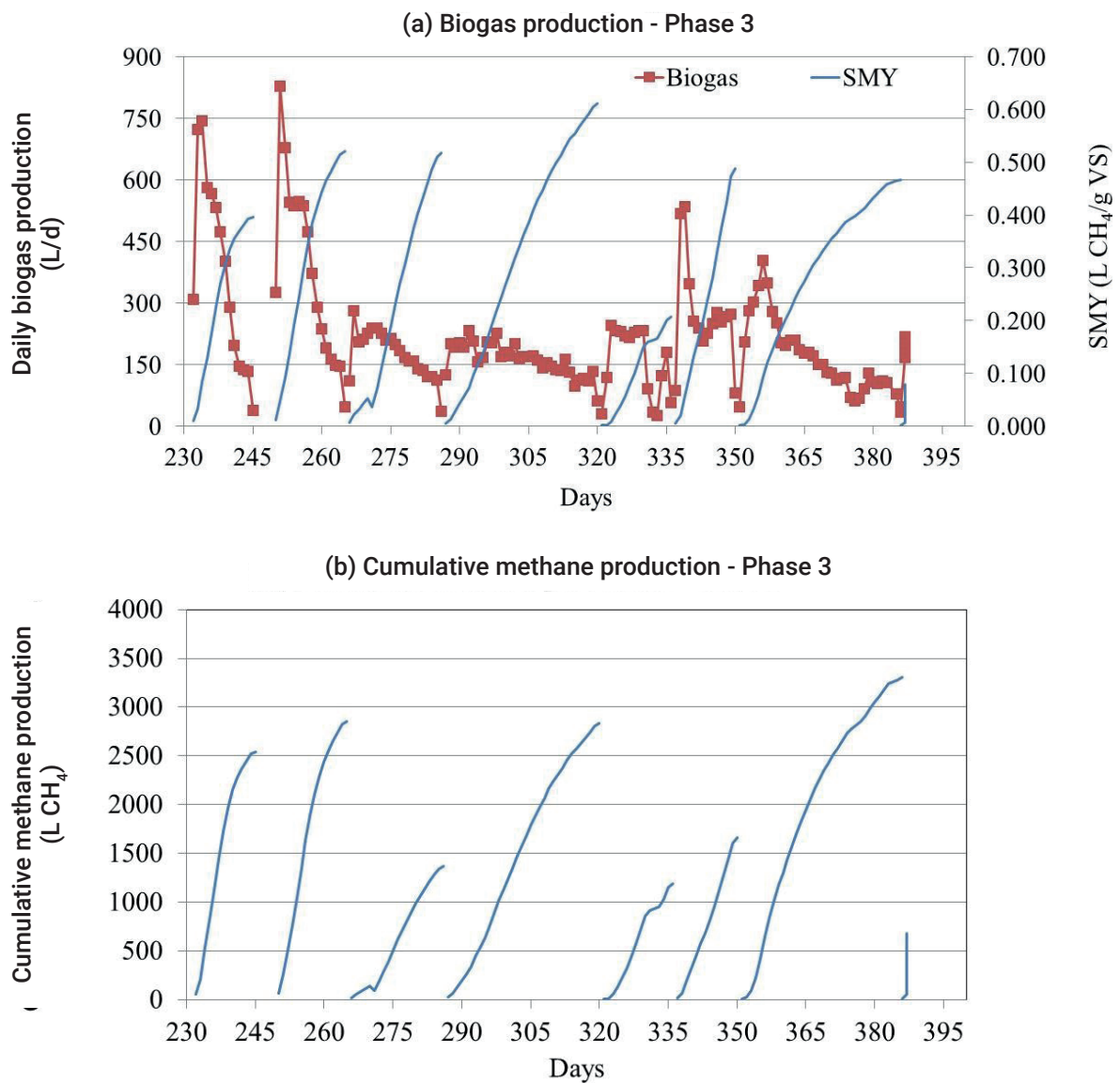


FIGURE 3: Performance of Phase-3 (high OLR) HSAD process: a) Biogas production and SMY evolution; b) Cumulative methane production.

cycles obtained only 44% reduction. This low conversion obtained was contradictory with the SMY measured using the biogas production, which indicates a proper organic fraction digestion. In that case, SMY would be considered more precisely since the biogas measurement was done on a continuous basis with instruments, while the mass balance was performed based on one-time sample taken on a large pile of heterogeneous material. Even though all precautions were taken to assure the proper sampling, it may be possible that the sample contains a piece or non-digested FW that contaminated the sample. Once again, the mass balance was not conclusive for the TS accumulation. Based on the values presented, the treatment cycle led to an accumulation of 44 kg. This value was not realistic as only 24 kg was provided from the VS accumulation and 5 kg was from the inorganic fraction of solids. It is not evident that that all the remaining 15 kg was contributed by the liquid inoculum (at 1.5 to 2% TS). Similar to the previ-

ous cycle of operation, the presence of inert material could explain the portion of the solid accumulation measured in the mass balance.

The total organic mass increase was about 88% of the total feed weight. The majority of the weight loss was not due to the VS degradation but from the water content released throughout the digestion process. Similar to the previous cycles, with a ~90% water content, the feedstock brings great quantity of water, which was released during the digestion process. From the mass balance, 503 kg of water was included in the feedstock. From that, only ~49 kg was taken out of the inoculum in 2 cycles (liquid inoculum production and water vapor in the biogas). About 447 kg of water was accumulated in the solid inoculum. According to the data, another 15 kg of water (3.0% of all water) was missing from the balance. The water accumulation in the solid inoculum was challenging and this showed an indicative of a lack of structure in the solid.

TABLE 2: Mass balance values for the low loading conditions (Inoculum to feed ratio of 3:1).

Cycle #	Inoculum start weight	Feedstock addition	TS fed	VS fed	Inoculum end weight	Inoculum TS in	Inoculum TS out	Inoculum VS in	Inoculum VS out
5 ^a	816 kg	279 kg	26 kg	24 kg	1 017 kg	138 kg	154 kg	89 kg	99 kg
6 ^b	1 205 kg	280 kg	21 kg	19 kg	1 495 kg	181 kg	209 kg	118 kg	132 kg
7 ^c	559 kg	167 kg	15 kg	14 kg	588 kg	123 kg	114 kg	92 kg	84 kg
8	568 kg	232 kg	20 kg	18 kg	559 kg	111 kg	109 kg	82 kg	75 kg
9 ^d	1 039 kg	368 kg	37 kg	32 kg	1 196 kg	188 kg	208 kg	136 kg	158 kg
10	1 071 kg	336 kg	21 kg	18 kg	1 381 kg	187 kg	220 kg	141 kg	163 kg
Total	5 258 kg	1 662 kg	140 kg	125 kg	6 236 kg	876 kg	1 014 kg	610 kg	711 kg
Average	876 kg	277 kg	23 kg	21 kg	1 039 kg	146 kg	169 kg	102 kg	119 kg

a: Cycle 5 was done with 1/3 of the available inoculum. 2/3 remaining was used for the low loading testing.

b: Cycle 6 was done with 2/3 of the available inoculum. 1/3 remaining was used for the 1:1 loading testing.

c: Cycle 7 had 62 kg of straw added (not included in the solid inoculum) adding 52 kg of TS, 48 kg of VS to the initial inoculum.

d: Cycle 9 feeding was done with 28.5% w/w of dairy manure and the rest was food waste.

Due to this reason, the further experiments were supplemented with more structural material and the optimisation study was carried out with this modifications. Structural agent (100 kg of dry straw) was used to increase the solid content and the draining capacity of the solid inoculum. The addition of straw increased considerably the volume of the solid inoculum and hence a small portion of initial solid inoculum was used. Wasted food residues was then fed to the newly mixed inoculum and it is to be noted that the straw weight was not considered in the feeding ratio calculation. The biogas production obtained was very different from the previous cycles. Starting at day 266, this cycle produced a small initial biogas production peak and maintained its production level for a longer period of time. It took about additional 2 days to reach the same conversion factor as the previous cycles. Reaction period was increased to 21 days to allow better adaptation to the straw addition.

On the contrary, cycle 8 (day 287) did not perform well as expected. However, the liquid inoculum percolation was good, henceforth no additional structural agent was added thereafter to the solid inoculum. It took 25 days to reach the expected conversion factor of 0.5 L_{CH₄}/g_{VS} fed. This delay in the biological conversion of organic solids to methane indicates the process imbalance. The hypothesis posed was that the C/N balance was too high with the addition of straw and nitrogen was lacking in the process. In order to help the biological process to resume its activity level, dairy manure was added to the feed material on cycle 9 (day 323). Dairy manure was used to rebalance the C/N ratio and the other required minerals for the digestion process. The total feeding ratio was kept around 3:1 (see Table 2) but 28.5% of the feeding weight was resulted from dairy manure (rest was FW). The addition of dairy manure and FW was treated in about 17 days, which was an improvement in comparison to 25 days of operation obtained previously.

The conversion factor reached was lower due to the lower biogas conversion potential of the dairy manure. With the fed proportion, the expected conversion factor to be reached was 0.33 L CH₄/g VS. The 10th cycle (day 337) was done using FVW alone and the operation went relative-

ly well by obtaining a biogas conversion rate of 0.487 L_{CH₄}/g_{VS} within 14 days. A significant biogas production peak was obtained for the first 4 days and then stabilised. Once again, the addition of low solid FVW dragged the solid inoculum to a low solid content (~15% TS). Structural agent may be needed for the next treatment cycle (Table 3).

Similar to the lower loading conditions, the biogas quality measured throughout the digestion process was highly variable mainly because of the operational procedures. Depending on a specific cycle, it took between 4 to 10 days to reach 55% of CH₄ in the measured biogas. If we consider that around 15-L of headspace was present in the laboratory scale bioreactor, we can use the dilution formula to estimate the biogas quality needed to bring the measured biogas composition at 55%:

$$C_1 V_1 + C_2 V_2 = C_3 V_3 \quad (1)$$

Where:

C₁: Methane concentration of the headspace

V₁: Volume of the headspace

C₂: Methane concentration of the produced biogas

V₂: Volume of biogas produced

C₃: Methane concentration measured

V₃: Volume of biogas and headspace

The calculated methane concentration is presented in Table 4. At the end of the treatment cycles, the methane concentration measured was between 54% and 63%. Soluble COD reduction of about 80-90% was obtained during this operation and TKN concentration of about 5-8 g N/L did not hinder the AD process. Except for the cycle 7, results indicate that adaptation of the biomass to the high loading conditions can be achieved but it may require longer reaction period. The impact of structural agent addition is yet to be fully understood.

The values obtained from the mass balance indicate that nearly 45% of TS and VS were degraded during the treatment cycles. A large fluctuation in the solids reductions were measured throughout the cycles. This high variation was probably due to the high heterogeneity level of the material making it hard to mix and sample representatively. Larger pieces of FW (e.g.: whole orange, potato,

TABLE 3: Values obtained for the high loading treatment cycle (3:1).

Cycle #	Inoculum: food ratio	OLR (g VS/Kg _{inoculum} /day)	Specific methane yield (SMY)* (L CH ₄ /g VS)
5	2.9 : 1	2.07	0.396
6	2.7 : 1	1.57	0.520
7	3.3 : 1	1.06	0.584
8	2.45 : 1	0.91	0.612
9**	2.82 : 1	1.87	0.321**
10	3.2 : 1	1.21	0.487

* The gas production cannot be measured accurately with the container type bioreactor. SMY was obtained from lab-scale bioreactor filled with the same material as the container.

** For this cycle, dairy manure was added with the FVW

TABLE 4: Calculated biogas composition during the first portion treatment cycle (Inoculum to feed ratio 3:1).

Cycle #	Days to reach 55% CH ₄ in biogas	Volume of biogas measured before 55%	Calculated % CH ₄ in first biogas	% CH ₄ at the end of treatment cycle
5	4	292.1 L	57.8%	60%
6	4	291.6 L	57.8%	63%
7	10	236.8 L	58.5%	54%
8	6	135.5 L	61.1%	63%
9	6	183.2 L	59.5%	62%
10	7	241.1 L	58.4%	62%
Average	6.2	230.05 L	58.9%	60.7%

avocado, corn, etc.) were harder to digest and some residual FW pieces were identified after the digestion process. TS and VS characterisation of the inoculum was then be greatly affected by the presence of those undigested materials.

A 45% VS reduction was considered a low value compared to the SMY measured with the biogas production. In the literature, FW degradation can be as high as 85% during anaerobic process leading to a SMY of 0.5 to 0.6 L_{CH₄} g_{VS}⁻¹. In this experience, SMY was considered more precisely since the biogas measurement was done on a continuous basis with precision instruments. Whereas the mass balance was done based on one sample taken in a large pile of heterogeneous material. Even though all precautions were taken to assure the proper sampling, the samples still contained small portion or pieces of non-digested FW.

During the high loading cycles, the total mass of the inoculum increased by 18.6% (TS accumulation of 138 kg) partially due to the structural agent addition and water retention. The average TS content of the solid inoculum was maintained above 15% by the addition of straw. FW material is generally a low solid content material that may not be a good source of feedstock for high solid content digestion if used as a sole source. Structural agent is necessary to maintain the high solid level in the bioreactor. During the test cycles, a total amount of 1 522 kg of water was fed with the FW to the digester. The majority of the feedstock water (840 kg) stayed in the solid inoculum (likely absorbed by the straw) while another 620 kg was released from the digester (liquid inoculum and water vapor in the biogas). The liquid inoculum can be used for further treatment cycle but excess will have to be used.

Further analyses are needed to establish the nutrient content of the liquid inoculum, which is required to establish its fertilizer potential.

3.4 HSAD technology: Final discussion and concluding remarks

The biogas production, especially in terms of SMY and CH₄ concentration in biogas, yielded from the low-to-moderate temperature HSAD in this study were found to be similar or marginally higher than that of mesophilic/ thermophilic AD. For instance, Zhang et al. (2012) obtained a greater digester stability while codigesting FW with cattle slurry, in 1:4 ratio, at OLR of 2 kgVS m⁻³ d⁻¹ in mesophilic conditions (36°C). At similar OLR and temperature, Agyeman and Tao (2014) determined the effects of FW particle size on co-digestion with dairy manure, in 1:1 ratio, in which they obtained a SMY up to 0.47 L_{CH₄} g⁻¹VS_{fed} (for coarse-grinded FW). Alternatively at thermophilic conditions (55°C), Castrillón et al. (2013) used lower proportions of FW (i.e. 10% FW and 90% CM) to obtain the stability, which corresponded to a SMY of 0.3 L_{CH₄} g⁻¹VS_{fed}. In the present study, lowering the temperature to 20-25°C assured a good SMY and operational stability even for the mono-digestion of FVW. The reason could probably be due to that fact that at lower temperature, reduced hydrolysis of complex organics have declined the acidogenesis and thus reduced the proportion of CO₂ in biogas and additional production of acetate from CO₂ and H₂ by homoacetogens and the decrease of the resulting acetate would upsurge the amount of CH₄ in biogas (Wei et al., 2014). It is to be noted that low temperature AD is particularly well adapted to the treatment of several organic wastes because of lower free ammonia nitrogen

concentrations than in mesophilic/thermophilic AD processes. In addition to this, the present study also validated that solid CM can be used as a co-substrate provided a sufficient buffering capacity to FVW digestion by synergizing the effect of microorganisms and handling the high OLR. Nevertheless, further optimization will be essential to validate and improve the performance of HSAD at relatively short cycle length and high OLR.

In order to implement this HSAD technology for larger scale operations, economic cost analysis is essential for the financial success. The amount of methane produced in this process can directly influence the economic benefits, as this can substitute other fuels used for cooking, heat, light, or electricity. For example, it is critical to have methane concentration greater than 50% and H₂S concentration less than 1% for running a generator fueled with biogas (Lansing et al., 2008). In this study, HSAD technology met the minimum conditions to power a generator and coupled with the quantity of methane produced. Methane content of 65% in biogas has an overall energy potential of approximately 23 MJ m⁻³. According to U.S EPA (2016), over 38.4 million tons of US food waste generation was reported in 2014. Since food makes up over 20% of municipal solid waste combusted with energy recovery or landfilled in US, 12 commercial food disposal bans are often seen as a precarious step towards the long-term waste reduction goals (U.S. EPA, 2016). Keeping in mind that the calorific value of biogas is in the range of 6 kWh m⁻³ (which is equivalent to 0.5 L of diesel oil) (Kashyap et al., 2003), the proposed low-to-moderate temperature HSAD process could lead to saving of an enormous amount of fuel per year. Furthermore, by trapping methane from municipal or agricultural wastes for heat or electricity also diminishes direct atmospheric methane emissions and with it, greenhouse gas impact. For instance, diverting one ton of FW through HSAD reduces greenhouse gas emissions by nearly one ton of CO₂ equivalents, as compared to landfilling (Environment Canada, 2013). Similarly, Canadian livestock produces about 180 MT of manure every year. Treating 20% of manure would lead to a profit of \$55M in terms of carbon credit and bioenergy generated can replace electricity/natural gas to a value close to \$100M per year. Thus, the asset of this low-to-moderate temperature HSAD technology is that it provides an integrated solution to municipal and agricultural waste streams without any dilutions.

4. CONCLUSIONS

This study validated the robustness of low-to-moderate temperature HSAD technology, which can be employed to treat high-solid content wastes such as dairy manure and FW. S_{CH₄} of 0.4-0.6 L_{CH₄} g_{VS}⁻¹ was obtained even at high OLR (1.6 to 2.1 kg_{VS} kg_{inoculum}⁻¹ d⁻¹) and short cycle length (14-16 d), which is comparable to the laboratory scale study. Lowering the temperature to 20-25°C even favoured the mono-digestion of FVW. The mode of operation (process, temperature) along with the acclimation of liquid biomass to solid inoculum at step-wise increase in OLR ensured a high stabilisation of the digestion process without inhibi-

tion. Compared to higher-temperature digestion process, more energy is available for farm uses and thus farmers could adopt this technology at affordable cost. Further research is being performed to determine the optimal operating conditions.

ACKNOWLEDGEMENT

Authors thank Agri Innovation Program (Project No. 103) for providing financial support.

REFERENCES

- Agyeman, F.O., Tao, W., 2014. Anaerobic co-digestion of food waste and dairy manure: effects of food waste particle size and organic loading rate. *J. Environ. Manage.* 133, 268–274.
- APHA., 1992. Standard methods for the examination of water and waste water. 18th ed. Washington DC, USA: American Public Health Association.
- Braguglia, C.M., Gallipoli, A., Gianico, A., Pagliaccia, P., 2018. Anaerobic bioconversion of food waste into energy: a critical review. *Bioresour. Technol.* 248, 37–56.
- Castrillón, L., Marañón, E., Fernández-Nava, Y., Ormaechea, P., Quiroga, G., 2013. Thermophilic co-digestion of cattle manure and food waste supplemented with crude glycerin in induced bed reactor (IBR). *Bioresour. Technol.* 136, 73–77.
- Environment Canada., 2013. Technical Document on Municipal Solid Waste Organics Processing. ISBN:978-1-100-21707-9. (Viewed online on September 2017). http://www.compost.org/English/PDF/Technical_Document_MSW_Organics_Processing_2013.pdf
- Kashyap, D.R., Dadhich, K.S., Sharma, S.K., 2003. Biomethanation under psychrophilic conditions: a review. *Bioresour. Technol.* 87, 147–53.
- Khairuddin, N., Manaf, L.A., Halimoon, N., Ghani, W.A.W.A.K., Hassan, M.A., 2015. High solid anaerobic co-digestion of household organic waste with cow manure. *Procedia Environ.Sci.* 30, 174–179.
- Lansing, S., Botero, R.B., Martin, J.F., 2008. Waste treatment and biogas quality in small-scale agricultural digesters. *Bioresour. Technol.* 99 (13), 5881-5890.
- Massé, D.I., Saady, N.M.C., Rajagopal, R., 2014. Psychrophilic dry anaerobic digestion of high solids content dairy manure: long-term operation. *Biological Engineering Transactions* 7(3): 99-112.
- Rajagopal, R., Massé, D.I., Saady, N.M.C., 2014. Low-temperature anaerobic co-digestion of swine carcass and swine manure: Impact of high swine carcass loading rate. *Transactions of the ASABE* 57 (6), 1811-1816.
- Rajagopal R, Bellavance D, Rahaman S., 2017. Psychrophilic anaerobic digestion of semi-dry mixed municipal food waste: For North American Context. *Process Safety and Environmental Protection* 105, 101-108.
- Saady, N.M.C., Massé, D.I., 2015. A start-up of psychrophilic anaerobic sequence batch reactor digesting a 35% total solids feed of dairy manure and wheat straw. *AMB Expr* 5:55.
- U.S. EPA (Environmental Protection Agency), 2016. Food Waste Management in the United States, 2014. https://www.epa.gov/sites/production/files/2016-12/documents/food_waste_management_2014_12082016_508.pdf (accessed 10/11/2017)
- Wei, S., Zhang, H., Cai, X., Xu, J., Fang, J., Liu, H., 2014. Psychrophilic anaerobic co-digestion of highland barley straw with two animal manures at high altitude for enhancing biogas production. *Energ. Convers. Manage.* 88, 40-48.
- Yang, L., Huang, Y., Zhao, M., 2015. Enhancing biogas generation performance from food wastes by high-solids thermophilic anaerobic digestion: effect of pH adjustment. *Int. Biodeterior. Biodegrad.* 105, 153–159.
- Zhang, Y., Banks, C.J., Heaven, S., 2012. Anaerobic digestion of two biodegradable municipal waste streams. *J. Environ. Manage.* 104, 166-174.
- Zheng, Y.H., Wei, J.G., Li, J., Feng, S.F., Li, Z.F., Jiang, G.M., Lucas, M., Wu, G.L., Ning, T.Y., 2012. Anaerobic fermentation technology increases biomass energy use efficiency in crop residue utilization and biogas production. *Renew. Sust Energy Rev* 16: 4588–4596.