Thermophilic anaerobic digestion of the screened solid fraction of dairy manure in a solid-phase percolating reactor system

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Abstract

An increase in volatile solids (VS) content from solid fraction of dairy manure would reduce the size of biogas on-farm facilities and could be of greater interest to farmers than manure slurry biogas plants. This study examined the technical feasibility of a solid-phase batch thermophilic anaerobic digestion system for the screened solid fraction (SF) of dairy manure using the digested liquid fraction of dairy manure as inoculum. Inoculum to substrate ratio (I/S) and percolate recirculation strategy were the parameters studied. The manure slurry separation process resulted in a solid fraction that represented 16.8% of total manure mass; this aforementioned fraction showed an ultimate methane yield of 61.5 L CH_4 kg^{-1} SF (265 L CH_4 kg^{-1} VS) which represented 48% of methane potential from raw manure slurry. Five comparative experiments with 40 kg SF and different I/S ratios and percolate recirculation rates revealed that higher I/S ratios provide more security against inhibition states due to high VFA levels and low pH in percolate. The results of the present work have shown that increasing the percolate recirculation rate in intermittent and short recirculation operations improves the stability and speed of the process. Under this percolate recirculation strategy dry batch operation was possible with a I/S ratio of 0.6 obtaining methane yields of 145, 175, 204 and 220 L CH_4 kg^{-1} VS after 15, 20, 30 and 60 days of operation.

Keywords: Solid-phase anaerobic digestion, dairy manure solid fraction, biogas, percolate, inoculum.

1. Introduction

The increase in production and concentration of intensive livestock operations have resulted in greater awareness and concern for the proper management of livestock manure. In Cantabria (a small region on the Northern Coast of Spain), as in the majority of western European countries, livestock farming has become more specialized leading to a reduction in the number of farms. This reduction in the number of farms has been accompanied by an increase in the number of animals per farm. For instance, the average size of dairy farms in Cantabria increased 72.5% between 1995 and 2010. The significant growth in farm size has contributed to a host of environmental problems (Billen et al., 2015). Storage and land spreading, the traditional dairy manure treatment, remain the only manure management strategy in Cantabria. It allows for the emission of ammonia, particulate matter, unpleasant odours, volatile organic compounds and a variety of other air pollutants (Brito et al., 2012). Intensive dairy farms annually produce thousands of tons of manure that can spill into waterways from leaking manure pits and lagoons or fields where manure is over-applied as fertilizer. Moreover, while in storage the submerged manure generates methane, a greenhouse gas (GHG) with 21 times the global warming potential of carbon dioxide, according to the Intergovernmental Panel on Climate Change (IPCC).

Anaerobic digestion is a well-known technological process that constitutes an alternative to traditional dairy manure management practices (Thu et al., 2012; Nasir et al., 2012). Anaerobic digestion not only alleviates environmental concerns, but also converts manure into two categories of valuable products: biogas, a renewable fuel and the digested manure with improved fertilizer characteristics (Holm-Nielsen et al., 2009). In addition, recovery of manure biogas is an acknowledged cost-effective mitigation technology for GHG emissions in agriculture (Hamelin et al., 2014). However, farmers often use liquid manure management systems that use water to flush or clean alleyways or pits where the manure is excreted. As a result dairy manure can be too diluted resulting in low volatile solids (VS) content and low methane yield which makes conventional slurry digestion systems not very effective (Demirer and Chen, 2008). In this regard Asam et al. (2011) reported that animal slurries with water content higher than 90% make the economic viability of biogas plants difficult.

One method to obtain fractions of manure with higher biogas potential in terms of volume is by increasing VS content, which can be achieved by means of solid-liquid separation (Møller et al., 2004). In fact, separation of manure into liquid and solid fractions is a treatment option that complements anaerobic digestion. It can be used as a pre-treatment to obtain concentrated solid fractions with higher methane yields or as post-treatment to obtain a nutrient-rich solid fraction that reduces the cost of transportation (Møller et al., 2002). In this regard, Møller et al. (2007) and Sutaryo et al. (2012) studied the process performance of manure based digesters using the solid fraction of manure as co-substrate. Hamelin et al. (2011) postulated this new biogas production concept as a probable scenario for future biogas production due to limitations in availability of other biomasses such as organic wastes and energy crops. The microbiologically produced hydrogen (biohydrogen) is also a possible future competitor for conventional anaerobic digestion of lignocellulosic biomass (Kumar et al., 2015) due to its inherent advantages, such as relatively low energy demand, the usability of a wide range of feedstocks, and the possibility to integrate with other biological processes to enhance biohydrogen or biogas upgrading (Bakonyi et al., 2014; Luo and Angelidaki, 2013).

Under this scenario anaerobic digestion of the solid fraction of dairy manure could be a suitable alternative for intensive dairy farms in Cantabria. Dry, high-solids or solid-phase anaerobic digestion is a relatively new application of anaerobic treatment processes in the agricultural sector (Weiland, 2006). There is no clear dividing line between the terms wet and dry digestion but it is assumed that dry digestion is referred to for substrates with total solids (TS) content higher than 20% (Abbassi-Guendouz et al., 2012). The dry anaerobic digestion process can be performed under batch, continuous or semi-continuous mode of operation (Karthikeyan and Visvanathan, 2013). Batch systems for dry digestion processes are emerging because they require less process control measures than the continuous processes and they are less susceptible to failure (Karthikeyan and Visvanathan, 2013).

The interest in dry batch anaerobic technologies for solid manure in batch leaching bed reactors is rising due to its advantages compared with wet anaerobic digestion systems in CSTR digesters, such as: higher methane yields per mass unit of substrate, lower amount of water needed for the digestion process, the reduced amount of digestate

generated and the reduced size of reactor needed (Demiren and Chen 2008; Fierro et al., 2014). On the other hand, several drawbacks have been reported for solid manure dry anaerobic digestion systems such as long degradation times, low methane conversion yield, high inoculation rate and high sensitivity to inhibition if the water content is low (Shewani et al., 2015). In leaching bed reactors, solid manure remains stationary throughout the process and contact with microorganisms is enhanced by recirculation of a percolation liquid (percolate, leachate) which is sprayed over the organic matter in the digester. During the digestion process no moving parts, such as mechanical stirrers or screw conveyor feeders, are required, thus resulting in low system operation and maintenance costs. As a disadvantage, mass transfer properties are limited. Although the basic technology of dry digestion for substrates such as the organic fraction of municipal solid wastes (OFMSW), food and vegetable wastes and energy crops is well implemented (Karthikeyan and Visvanathan, 2013) and despite the potential advantages of dry anaerobic systems for animal manure, little information is available about dry digestion using solid cow manure as substrate: Hall et al. (1985) utilized a packed bed digester for the mesophilic anaerobic digestion of solid cattle waste; El Mashad et al. (2006) studied the effect of inoculum addition modes and leachate recirculation on the anaerobic digestion of solid cattle manure in an accumulation system; Demirer and Chen (2008) developed a leaching bed reactor packet with a mixture of dairy manure, anaerobic seed, wood chips and water to generate a leachate; Massé and Saady (2015) studied the psychrophilic dry anaerobic digestion of dairy cow faeces.

To the best of the author's knowledge, no previous reports are available in the literature about dry anaerobic digestion of the solid fraction of dairy manure in a solid-phase percolating reactor system. The use of the digested liquid fraction as liquid inoculum is also a new approach. The aim of the present work was to develop a solid-phase percolating anaerobic digestion system for the solid fraction of dairy manure. In the proposed system process, neither water nor bulking material was added. The digested liquid fraction of dairy manure has been used as inoculum and the following aspects have been addressed: technical viability of the process, influence of inoculum to solid substrate ratio and percolate recirculation strategy.

2. Materials and Methods

2.1. Solid fraction of dairy manure and inoculum

The solid fraction (SF) of dairy manure used as substrate in the solid-phase anaerobic digestion process was collected from a 600-free stall dairy cow farm, located in Loredo (Cantabria). The farm is equipped with scrape systems and manure pit storage. Raw manure slurry was separated into solid and liquid fractions by means of a screw press separator (Cri-Man SM260-75, 0.75 mm mesh). The screened liquid fraction (LF) of dairy manure was digested at 50ºC and used as inoculum (I) for the solid-phase anaerobic process.

2.2. Mass Balance and BMP test

The characteristics of the raw manure, LF and SF are presented in Table 1 (mean values during the experimentation). A mass balance was calculated from distribution of TS with the aim to determine the percentage of manure methane potential that can be obtained from the solid fraction.

A biochemical methane potential (BMP) test was carried out to determine the methane potential from the manure, LF and SF. The test was performed in duplicate using 500-mL serum bottles capped with rubber septum sleeve stoppers as reactors. The digested liquid fraction of the manure was used as inoculum in the BMP tests. The inoculum to substrate ratio was 1 in terms of VS. For the manure and the LF the test was performed at 35ºC whereas for the SF the test was carried out at 50ºC. Gas production was determined by pressure measurement. Biogas samples were taken through the septum by a needle connected to a syringe to analyse its methane content. All the reactors were manually stirred once a day. After the set-up of the reactors, helium was flushed to remove the air in the headspace of the bottles. The duration of the BMP test was 80 days. Two blanks with water and inoculum were also tested to measure methane potential of inoculum. Results are expressed as means subtracting methane production from the blanks.

2.3. Solid-phase batch anaerobic digestion system

2.3.1. Solid-phase anaerobic digester

Batch experiments were conducted in duplicate in two identical vertical cylinder-shaped tanks made of 304 stainless steel, adapted as dry anaerobic digesters. The dimensions of each reactor were 40 cm in internal diameter and 100 cm in height with an operating volume of 90 L. Each digester was divided into three separable parts joined by flanges to facilitate cleaning after operation. The inoculum-percolate distribution device was placed in the upper part of the digester. It consisted of a tube, the end of which was submerged in a small glass-shaped vessel that, by overflowing, allowed the distribution of the percolate over the surface of the solid substrate through a perforated plate (36 holes, 5 mm diameter). The digesters were equipped with pressure sensors (ifm, PI-16789, 0-100 mbar) and temperature probes (bimetallic thermometer, 60 cm stem length, 0-80ºC). Biogas production was measured by means of home-made drum-type biogas meter devices. Stable reactor temperature was maintained at 50ºC in the digesters by means of electric heating blankets that covered the external surface of the central part of the digesters. In addition, the digesters were thermally insulated with a 2-cm layer of rubber foam. The middle and the lower parts were separated by a perforated metal plate (33 holes, 10 mm diameter) that allowed the separation and collection of the percolate in the lower part of digester, used as percolate storage tank with a capacity of 10 L. A scheme of the experimental solid-phase batch digestion process is shown in Fig. 1.

2.3.2. Digester operation

Previous to dry batch digestion operation, 160 L of the LF were digested at 50ºC to be used as inoculum for the subsequent solid-phase percolating reactor system. Manual recirculation was selected as the most suitable option for the system process at the projected scale. Centrifugal pumping created too much suction, which washed down solids with the percolate, resulting in blockage problems in the pipeline. On the contrary peristaltic pumping resulted in poor distribution of the percolate over the solid substrate surface. Manual recirculation avoided solids washing and blockage problems and allowed a very good distribution of the percolate over the solid substrate surface. At the end of the retention time the percolate was drained and collected to be re-used as inoculum in the following cycle. For each experimental operation, the methane yield from liquid inoculum was also tested and taken into account to determine the methane

production from the solid substrate in the dry tests. In any case, methane production from inoculum was negligible compared to methane production from the solid substrate.

2.4. Experimental configuration

In the framework of this study, five duplicated solid-phase anaerobic digestion experiments with the solid fraction of dairy manure as substrate were carried out. The results showed are the mean values of duplicated experiments. For all the experimental runs the solid-phase digester was loaded with 40 kg of the SF and operated for 60 days. After loading the SF, the liquid inoculum was added through the percolate distribution system. Then, the heating system was connected and the digester started to run. Liquid inoculum was sieved through a 1 mm mesh sieve before the beginning of each experiment.

Besides technical feasibility, inoculum to solid substrate ratio (I/S) and percolate recirculation rates was studied. Operation with different amounts of liquid inoculum and recirculation rates were assayed. Table 2 summarizes the experimental set-up. During the first 24 hours of each experiment no recirculation of percolate was performed.

2.4.1. Experiment 1 (E1)

In order to study uncertainties about the I/S ratio, the first experiment was carried out adding 60 L of liquid inoculum (I/S ratios 1.5 in mass basis) and a very simple percolate recirculation strategy: 10 L were recirculated once a day. Recirculation of percolate was done manually extracting a litre from the bottom part of the reactor and re-introducing it through a funnel to the distribution system in the upper part which took about 1 minute. This operation was repeated ten times consecutively and lasted about 10 minutes.

2.4.2. Experiment 2 (E2)

The second experiment was carried out adding 30 L of liquid inoculum (I/S ratios 0.75 in mass basis). In this experiment the liquid inoculum employed was that previously used in E1. The percolate recirculation strategy was the same as that in E1.

2.4.3. Experiment 3 (E3)

Based on the results of the previous trials, the third experiment was carried out adding 40 L of liquid inoculum (I/S ratio: 1). The liquid inoculum employed was that previously used in E2. Percolate recirculation rates were increased: 30 L of inoculum per day were recirculated in three periods (10 L x 3 times) distributed throughout the day. From Monday to Friday recirculation was done manually. During the weekends a peristaltic pump (flow 1 L min^{-1}) was programmed to recirculate 30 L of percolate per day with a similar recirculation strategy to manual mode.

2.4.4. Experiment 4 (E4)

In experiment 4, the digester was also inoculated with 40 L of inoculum. The liquid inoculum employed was that previously used in E3. In this case 20 L of inoculum per day were recirculated, but distributed in ten operations throughout the day (2 L x 10 times). From Monday to Friday recirculation was done manually. During the weekends a peristaltic pump (flow 1 L min^{-1}) was programmed to recirculate 20 L of percolate per day with a similar recirculation strategy to manual mode.

2.4.5. Experiment 3 (E5)

The last experiment (E5) was performed with the lowest amount of inoculum: 24 L (I/S ratio: 0.6). Previous assays showed that the process was alkalinity consuming. For this reason, "fresh" inoculum (digested liquid fraction of dairy manure) was added to the digester. Percolate recirculation rates were increased: 30 L per day distributed in fifteen operations throughout the day (2 L x 15 times). During the weekends a peristaltic pump (flow 1 L min^{-1}) was programmed to recirculate 24 L of percolate per day with a similar recirculation strategy to manual mode.

2.5. Analytical methods

Volatile fatty acids (VFA) were determined using a HP6890 gas chromatograph (GC) fitted with a 2 m 1/8-in glass column, liquid phase 10% AT 1000, packed with the solid-support Chromosorb W-AW 80/100 mesh. Nitrogen was used as the carrier gas at a

flow rate of 14 mL/min, and a FID detector was installed. VFA concentrations are expressed in COD units. Biogas composition was assayed on a 2 m Poropak T column in a HP 6890 GC system with helium as the carrier gas at a flow rate of 15 mL/min with a TCD detector. Biogas and methane volumes are expressed at 0ºC and 1 atm in dry conditions. Total Solids (TS), Volatile Solids (VS), pH, Chemical Oxygen Demand (COD), total Kjeldahl nitrogen (TKN-N) and NH_4^+-N were performed according to Standard Methods (APHA, 1998). Bicarbonate alkalinity (BA) was determined by titration at a pH of 5.1 according to the method described by Anderson and Yang (1992). Data were analysed by analysis of variance procedure (ANOVA) followed by Tukey's means grouping test to determine whether there were statistically significant differences among the five experiments. The significance level was set at 5%.

3. Results and discussion

3.1. BMP tests: distribution of methane potential

As shown in Table 1, raw manure slurry with 7.2% VS content was separated into a LF with 4.0% VS content and a SF with 23.2% VS content. As expected, a solid fraction with much higher VS content than raw manure was obtained from the separation process. After 80 days the methane yield from the manure, the LF and the SF were 298, 343 and 265 L CH_4 kg^{-1} VS respectively. In Fig. 2, methane yields per mass of sample are presented, resulting in 21.5, 13.7 and 61.5 L CH_4 kg^{-1} sample for the manure, the LF and the SF respectively which means that solid fraction has a potential methane yield per kg 2.86 times higher than that of manure. To determine the percentage of manure methane potential that can be obtained from the solid fraction, the mass balance of the separation process must be considered. Through the separation process 0.168 kg of the SF and 0.832 kg of the LF were obtained from 1 kg of manure, which yielded 21.5 L CH_4 per kg. Taking into account the mass balance and individual methane yields, the methane potential that can be recovered from the SF can be calculated according to the following equation, where Y represents methane yields.

$$\%\, CH_{4-SF} = \frac{\%\, mass_{SF} \cdot Y_{SF}}{Y_{manure}}$$

From this calculation, the solid fraction separated showed the potential to produce 48% of methane potential in raw manure. It is very interesting to note that the solid fraction represented 16.8% of the initial manure mass because such a small percentage of mass has the potential to produce 48% of manure methane potential.

3.2. Solid-phase digester operation

The first objective of the present work was to determine the technical feasibility of the proposed process. The success of the process depended on the ability of the liquid inoculum to trickle through the whole solid substrate stack allowing the contact between microorganisms and the solid substrate without clogging the drainage system. During the first hours of each trial, while the digester had not reached the operating temperature of 50ºC, percolation of liquid inoculum was very limited. As digester temperature increased percolation of inoculum was enhanced. No clogging problems were observed during all the experimentation. Kusch et al. (2011) reported that liquid manure slurry was not suitable for percolation in dry batch anaerobic digestion systems due to its high viscosity. In this regard El-Mashad et al. (2006) observed increased leachate recirculation volumes during anaerobic digestion of solid cattle manure in an accumulation system at 50ºC compared to 40ºC, which was attributed to the lower viscosity of leachate at 50ºC. Screening and thermophilic conditions are suggested as the reasons for the good percolation characteristics of the liquid inoculum used in the present study.

3.2.1. Methane production

In Fig. 3 volumetric methane production rates and methane content in biogas for the five experiments are shown. As the digester reached operating temperature, the liquid inoculum stimulated a rapid start-up of the process and the methane content in the biogas started to grow from the beginning of the experiments, as Fig. 3c and 3d show. The evolution of the methane percentage in the biogas showed a similar trend in the five experiments. From the start-up the methane content in the biogas progressively increased reaching values higher than 50% within approximately five days for all the trials.

Daily methane production rates were divided into three periods and statistically analyzed. The start-up of the process followed a similar trend in the five experiments. During the first five days of the trials, daily methane production rates in the five experiments showed no significant differences. A second statistical analysis was done for the following period, 5-30 day, which showed significant differences amongst three groups: E1, E3 and E4 had no significant differences amongst them whereas E2 and E5 showed meaningful differences in the evolution of daily methane yields, as can be observed in Fig. 3a. In E2 daily methane yields were lower than those observed in the other experiments and methane production rates did not reach a clear peak. These data prove that methane production in E2 was slower compared to the other experiments, which may be attributable to a partial inhibition state. The low inoculum to substrate ratio could be the reason (I/S 0.75). However in the last experiment carried out (E5), the I/S ratio was the lowest of all (0.60) but the digestion process showed a better performance which was supported by the statistical analysis. From day 30 to the end of the experiment, no significant differences were observed amongst the five experiments through the analysis of variance.

Fig. 4a shows the cumulative methane yields of the five experiments. The curves from E1, E3 and E4 are similar and situated between the curves of E2 and E5. Methane yields reached after 60 days were quite similar in the five experiments: 220, 214, 226, 227 and 227 L CH_4 kg^{-1} VS for E1, E2, E3, E4 and E5 respectively. The differences between the different trials were the methane production rates. E5 was the fastest in methane production. On the contrary, E2 was the slowest in methane production. In Fig. 4b the evolution of the percentage of methane produced in the dry batch digester through experimentation with respect to the BMP value (265 L CH_4 kg^{-1} VS) is compared for E2 and E5. During the first 20 days the difference in cumulative methane between the E2 and E5 yield increased. During the second half of the retention time differences between both experiments diminished.

3.2.2. Evolution of volatile fatty acids and pH in percolate

As can be observed in Fig. 5a, VFAs in the percolate followed a similar trend to daily methane yields. Initially there was no presence of VFAs in liquid inoculum. As microbial activity was activated by temperature increase and contact between

microorganisms and substrate, the VFAs concentration in the percolate rapidly increased. After reaching a peak, the VFAs were gradually consumed and after 20 days there was no presence of them in E1, E3, E4 and E5. However, the sum of individual VFAs was higher in E2 than in the rest of trials with a maximum value of 13.6 g COD_{VFA} L^{-1} at day 5. The graphs in Fig. 5b and 5c clearly show that acetic acid was the dominant VFA followed by propionic acid as the second most dominant. Butyric acid was also detected whereas the remaining volatile fatty acids were present at negligible concentrations. Again, the lower I/S ratio could be suggested as the reason for the high VFA levels in E2. A higher amount of liquid inoculum provides dilution of potential inhibitory substances and more bicarbonate alkalinity for pH control. With the double amount of liquid inoculum (E1), the VFAs reached a highest value of 6.9 g COD_{VFA} L^{-1} in the percolate; whereas in E3 and E4 (40 L of inoculum), maximum VFA levels of 8.5 and 9.8 g COD_{VFA} L^{-1} were observed. However, in E5, with the lowest amount of inoculum (24 L), the maximum VFAs concentration in the percolate was 9.9 g COD_{VFA} L^{-1}. Although acetic acid is usually present in higher concentrations than other fatty acids during anaerobic digestion, propionic and butyric acids are more inhibitory to the methanogens (Ward et al., 2008). Moreover, Pullammanappallil et al. (2001) observed that propionic acid was an effect rather than a cause of inhibition of anaerobic processes. Furthermore, degradation of propionic and butyric acids is inhibited by acetic acid (Ahring and Westermann, 1988).

In this sense, from data in Fig. 5b and 5c the maximum levels of propionic acid in E2 and E5 were similar, 1.4 g L^{-1} in E2 and 1.7 g L^{-1} in E5, but its degradation was slower in E2. On the other hand acetic acid levels in E2 reached a maximum value of 9.2 g L^{-1} whereas in E5 the maximum concentration value of acetic acid was 5.7 g L^{-1}. So the higher values of acetic acid in E2 can be the reason for the slower degradation rates and daily methane yields experimented in E2. In fact, during E2, pH in the percolate reached values as low as 5.4, as can be observed in Fig. 6a. Whereas in the rest of the experiments percolate pH stayed over 6.0, during E2 pH values remained around 5.5 between days 3 and 10. Although the optimum pH of hydrolysis and acidogenesis has been reported as being between 5.5 and 6.5, the optimal pH of methanogenesis is around pH 7.0 and lower values can inhibit methanogenesis (Ward et al., 2008). In E2, the process was not completely inhibited, but in view of lower daily methane yields it can be suggested that the combination of low pH and high acetic acid levels

experimented in the percolate was the reason for the lower methane production rates in E2. In this regard, Angelidaki et al. (1993) reported that interaction between VFAs and pH may lead to an inhibited state where the process runs stably but with a lower methane yield. There was no evidence for ammonia inhibition since NH_4^+-N concentrations in the percolate ranged from 1.6 to 1.0 g L^{-1} during experimentation, as shown in Fig 6b, which are below inhibition limits (Chen et al., 2008).

3.2.3. Influence of percolate recirculation strategy

During batch solid anaerobic digestion, the high VS concentration can affect process efficiency due to inhibition phenomena (Massaccesi et al., 2013). In the present study the origin of this inhibition would be VFA or ammonia accumulation if they were not washed out frequently. By comparing E2 and E5, we can suggest that an adequate percolate recirculation rate was of great importance to manage potential inhibition states due to low pH and high VFAs, which made the process slower. Bicarbonate alkalinity from inoculum was the main buffer source for pH control. At the beginning of E2 there was 543 g $CaCO_3$ kg^{-1} VS_{SF} whereas in E5 less alkalinity was available: 454 g $CaCO_3$ kg^{-1} VS_{SF} (see Table 2). Even though E2 was performed with an initial higher amount of liquid inoculum and bicarbonate alkalinity, during the first days of operation the percolate pH rapidly dropped and maintained values of around 5.5 that resulted in lower daily methane yields compared to those of E5. The difference between both experiments was the percolate recirculation strategy. In E2, 10 L of percolate were recirculated in only one daily operation which would lead to VFA accumulation in the solid substrate and inhibition states mainly in the upper middle part of the substrate stack where dilution of VFA and mass transfer would be hindered due to limited water supplementation. In this regard Veeken and Hamelers (2000) reported only small amounts of methane production during dry batch anaerobic digestion of OFMSW without percolate recirculation. In E2, acidification was not irreversible. This can be explained by the fact that the bottom part of the solid substrate stack remained flooded and 10 L of daily recirculation was enough to keep the process ongoing, although at a low rate compared with the rest of the experiments. A comparison was made between the behavior observed in E2 and that of E5, in which 30 L d^{-1} of percolate was recirculated in fifteen 2-L recirculation operations distributed throughout the day. This comparison showed that the recirculation strategy applied in E5 allowed higher water

distribution and dilution of VFA through the whole substrate stack, which enabled an improved process operation performance.

The results from E1, where 60 L of liquid inoculum were used, showed that combination of higher amounts of inoculum and low percolate recirculation rates avoided acidification problems. This can be explained by the fact that the whole substrate stack was flooded, thus allowing mass transfer and VFA dilution. In experiments E3 and E4, where intermediate amounts of liquid inoculum were employed at different recirculation rates, we observed similar reactor performances. In this case the lower recirculation rates in E4 (20 L d^{-1}) compared with E3 (30 L d^{-1}) were compensated by a intermittent and short recirculation operation: 2 L in ten times versus 10 L in three times for E4 and E3 respectively.

3.2.4. Process considerations

Methane yields reached after 60 days in the dry process were about 15% lower compared to the methane yield of the SF obtained in the BMP test (265 L CH_4 kg^{-1} VS). After 60 days of operation, the dry batch process was still producing biogas, but at low rates. After the experiments, when the upper part of the reactor was withdrawn to remove the digested solid fraction, it was noted that the percolate distribution system created small holes on the surface of the solid that could create preferential channels in the stacked solid substrate mass. It suggests that despite having 36 holes for percolate distribution over the solid waste, contact between microorganisms and the substrate could be limited in some zones, which can explain the lower methane yield observed in comparison with BMP values. Benbelkacem et al. (2010) reported that appropriate leachate injection geometry is a key point to increase the overall process efficiency in leaching bed reactors. A sprinkler system would improve percolate distribution over the mass of solid waste improving the performance of the dry digester. With regards to HRT, 60 days is too long compared with typical HRT applied for manure slurries in CSTR systems, which can range from 15 to 30 days depending on manure characteristics and the operating temperature (Nasir et al., 2012). Reduced reactor size is one of the main advantages of the proposed process and HRT as high as 60 days would limit this advantage. Despite the factor that the percolate recirculation and distribution system was not the optimum, the methane yield in E2 after 30 days was

77% of the methane yield observed in the BMP test. The results obtained in this work suggest that higher methane yields could be obtained with lower HRT by improving percolate distribution (sprinkler system) and recirculation rates by automatic percolate recirculation pumping. In this sense, further investigation is necessary to determine the minimum I/S ratio that allows the success of the process and the optimum percolate recirculation strategy.

3.3. On-farm energy potential

An estimated 40 tons of dairy manure are daily produced on the farm that provided the solid fraction employed in this work. Using the BMP values obtained in this work to estimate the energy potential of the farm, biogas production from anaerobic digestion of 40 tons d^{-1} manure have the potential to run a 125 kW combined heat and power (CHP) system. Using only the solid fraction of manure with the solid-phase anaerobic system, 6.7 tons of SF per day have the potential to run a 60 kW CHP.

4. Conclusions

This study has demonstrated that thermophilic solid-phase batch anaerobic digestion of the screened solid fraction of dairy manure using the digested screened liquid fraction as inoculum is technically feasible. The process requires discontinuous recirculation of the liquid inoculum (percolate) to enhance contact between microorganisms and the solid substrate. The results of the present work have shown that increasing the percolate recirculation rate in intermittent and short recirculation operations improves the stability and speed of the process, allowing the operation with an I/S ratio of 0.6. The proposed process would allow intensive dairy farms to produce about 50% of methane potential from manure by processing the separated solid fraction, which in this work accounted only for 16.8% of manure mass. Thus, the size and related costs of the required facilities for a solid-phase anaerobic system could be more advantageous for farmers.

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Figure captions

Fig. 1. Experimental set-up scheme.

Fig. 2. Methane yield from manure, liquid and solid fractions.

Fig. 3. Evolution of volumetric methane production rates and methane content in biogas during the solid-phase batch process: (a) Volumetric methane production rates in E1, E2 and E5; (b) Volumetric methane production rates in E3 and E4; (c) Methane content in biogas in E1, E2 and E5; (d) Methane content in biogas in E3 and E4.

Fig. 4. Cumulative methane yields in solid-phase batch experiments: (a) specific methane yields for E1-E5; (b) Percentage of BMP yield reached in E2 and E5.

Fig. 5. (a) VFAs evolution in percolate during dry batch process; (b) Individual VFAs in percolate-E2; (c) Individual VFAs in percolate-E5.

Fig. 6. Evolution of percolate (a) pH and (b) NH_4^+-N concentration during solid-phase batch process.