



## Efficient biogas production from the liquid fraction of dairy manure

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**Abstract.** A farm integrated process for small scale biogas production from manure is designed. The anaerobic digestion (AD) process is tested on dairy manure and consists of a manure storage tank, a separation unit to remove coarse solids, and an AD reactor for biogas production using the resulting manure liquid fraction as substrate. The performance of the 220 L pilot AD reactor, run at 8.8 days hydraulic retention time depended on the raw manure storage time. The biogas production was 0.7-1.5 g COD L<sup>-1</sup> reactor d<sup>-1</sup>, and the maximum biogas methane yield was 0.19 g COD g<sup>-1</sup> feed COD. The overall AD process gained 29 % of the total methane potential of the raw dairy manure. High manure alkalinity (8-9 g CaCO<sub>3</sub> L<sup>-1</sup>) ensures stable pH (7.8-8.0) at high load. The results imply that dairy manure can be treated more sustainably by AD at even higher loadings.

### Key words

Biogas; Anaerobic digestion; Dairy manure; Manure pre-treatment; Manure storage.

### 1. Introduction

In order to reduce the greenhouse gas emissions from agriculture, the Norwegian Government has set a goal that 30 % of the manure is to be treated by anaerobic digestion (AD) within 2020. The use of AD also represents a source of alternative renewable energy in the form of biogas. According to a survey by Raadal [1] the biogas potential from wet organic wastes in Norway is nearly 6 TWh, of which 42 % is from manure. The costs of conventional AD plants treating manure are generally larger than the value of the products, due to low energy prices [2]. Few legal and economic instruments have been used in Norway to promote biogas production, and only very few biogas production plants based on agriculture substrates have therefore been built [2]. Biogas production must be cost-effective in order to achieve the goals proposed by the Norwegian Government. Cost-effective biogas production may be achieved by high rate anaerobic digestion (HRAD) reactors which require low reactor

volumes [3]. HRAD reactors like UASB (upflow anaerobic sludge blanket) can be fifty times more efficient than traditional continuous flow stirred tank reactors (CSTR) used in agriculture and wastewater sludge treatment [4]. The technological breakthrough of HRAD reactors has led to an exponential growth of the application of UASB and other sludge bed reactors worldwide. There is however no cheap alternative for the treatment of manure [3] because high rate reactors require a feed with low particle content whereas manure contains a variety of particle sizes in large quantities. The solid fraction must therefore be separated from the waste before feeding the liquid fraction to a high efficiency reactor. Use of solids “separator-liquefier-acidifier” has been tested in lab-scale reactors [5-6], also using polymers as flocculants before separation of the manure [7-8] but not extensively implemented.

The intention of the present study is to investigate if utilizing existing farm infrastructure and separation by a low cost sieve to limit investment and operational costs is a feasible strategy for Norwegian farms. To test this in pilot scale the designed process combining manure storage, particle separation and HRAD for biogas production is tested on a dairy farm. Norwegian dairy farms all have storage facilities for at least 8 months manure storage capacity to comply with government regulation that manure can only be spread on the fields during the 4 months growth season. These large storage facilities are proposed to be used as feed storage tanks for HRAD in which the first degradation steps (e.g. hydrolysis) will occur. Manure from 60 dairy cows producing organic milk at Foss farm was stored under different conditions before used as feed for an AD reactor. The biogas yield, biogas production rate and manure storage effects were examined. The HRT was kept constant at 8.8 days during the whole experiment. Lower HRT is obviously required to obtain truly HRAD and the benefits from such, but this is a topic for future studies if the present study confirms the hypothesis that the process scheme investigated can be used to achieve sustainable biogas generation from manure.

## 2. Materials and methods

The process scheme investigated contains storage of dairy manure in storage tanks, sieving of the manure to remove larger particles followed by digestion of the liquid fraction in a sludge blanket reactor.

### A. Manure storage

The cows are fed 25 % dairy concentrate (19 % protein) and the rest is grass/clover ensilage. The dairy manure was stored under different conditions as listed in Table 1 before used as feed for the AD reactor. The manure feed was a mixture of manure of different storage time related to the farms seasonal manure handling procedures and the project aim of testing the influence of manure storage

history. The manure handling includes flushing the manure into an indoor temporarily tank using small quantities of water (diluting the manure by 14 % on average). During the experiment the manure was pumped every third week (fourth from 15.May to 15.September) from the indoor temporarily tank to an open 1200 m<sup>3</sup> outdoor storage tank. The experiment started early summer (3. June) and lasted to 18.October. The manure storage history tested (Table 1) is summarized as follows: It was taken from the 1200 m<sup>3</sup> outdoor storage tank the first 95 days and last 14 days (period A, B and E). Manure stored 0-30 days in the indoor temporarily storage was used in period C (days 96-108), while during period D (days 109-122) the manure was collected behind the cows in the barn (no storage).

Table 1. Manure storage conditions during the experiment.

Period	Time (d)	Storage place	Storage conditions
A and B	0-60 and 61-95	1200 m <sup>3</sup> open outdoor storage tank	Stored during winter (ambient temperatures below zero), slowly increasing in temperature reaching summer (ambient temperatures 15-25 °C in summer). Partly mixed with fresh manure every 4 weeks. 1/2 of manure volume in the tank removed to fertilize fields during the first 30 days of the experiment.
C	96-108	Indoor closed temporarily tank	Fresh manure, stored 0-30 days.
D	109-122	–	Fresh manure, not stored.
E	123-137	1200 m <sup>3</sup> open outdoor storage tank	Stored outside. Contain a mixture of manure produced during winter and summer. Partly mixed with fresh manure every 3-4 week. Decreasing temperature during autumn.

### B. Pilot AD reactor system

The total process consisted of a 400 litre feed tank filled from sources as described in Table 1, a vacuum separation device made from a sieve with 1.4 mm mesh openings and an AD reactor (Figure 1).

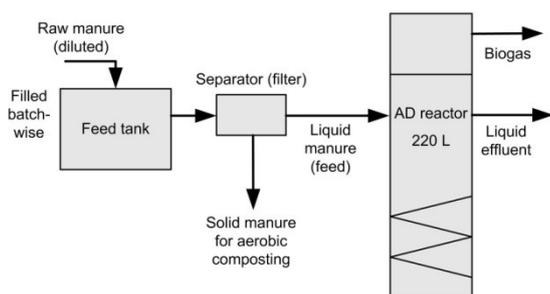


Fig 1. Pilot AD reactor process treating dairy manure (Aerobic composting of solid manure is not investigated in the present study).

The pilot AD reactor was built from a 2 m long, 0.4 m diameter PVC tube with top and bottom sealed off with standard end fittings. It has a liquid volume of 220 L and contains connected internal and external circulation loops made of 20 mm diameter PVC pipes (Figure 2). The reactor was seeded with 20 L unsieved dairy manure and operated in the psychrophilic and mesophilic ranges with a

start up at 25 °C and change to 37 °C on day 41. The HRT was kept constant at 8.8 days during the whole experiment.

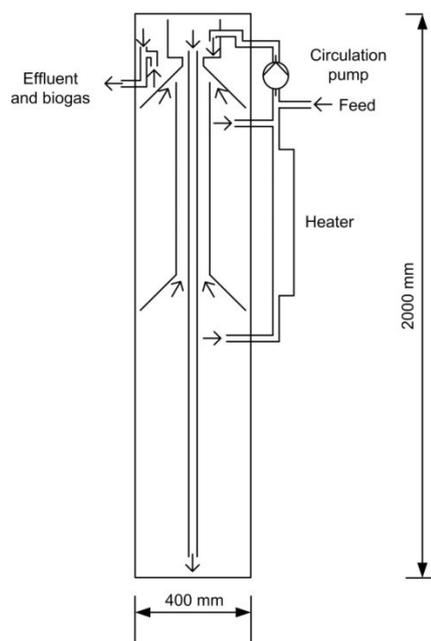


Fig. 2. Flow diagram of the pilot AD reactor.

### C. Monitoring and analysis

Gas production ( $L d^{-1}$ ) and reactor temperature were monitored continuously. Biogas, inflow and outflow liquid samples were collected weekly. Total COD ( $COD_T$ ), soluble COD ( $COD_S$ ), total solids (TS), volatile solids (VS), total suspended solids (TSS), volatile suspended solids (VSS), pH, alkalinity,  $NH_4^+-N$ , VFA's (acetate, propionate, butyrate, iso-butyrate) and gas composition ( $CO_2$  and  $CH_4$ ) were analyzed. The biogas flow was measured online using a volumetric gas meter working according to the same principles as used by Dinamarca and Bakke [9]. The temperature in the reactor was measured by a (PT100) sensor placed in the reactor wall at 1 m height. COD was measured according to US standard 5220D [10]. The samples were filtered ( $0.45 \mu m$ ) before  $COD_S$  determination. Alkalinity was measured by titration according to US standard 2320B [10].  $NH_4^+$  concentration was analyzed on filtered samples ( $0.2 \mu m$ ) by ion chromatography. DX-500 ion chromatographic analyzer equipped with a conductivity detector, a SCS1 cation-exchange column ( $4 \times 250 mm$ ) in combination with a Dionex IonPac PCG1 ( $4 \times 50 mm$ ) guard column. 4 mM methane-sulfonic acid was used as the mobile phase. The oven temperature was kept constant at  $35^\circ C$ . VFA's were measured by gas chromatography (Hewlett Packard 6890) with a flame ionization detector and a capillary column (FFAP 30 m, inner diameter  $0.250 mm$ , film  $0.5 \mu m$ ). The oven was programmed to go from  $100^\circ C$ , hold for one minute, to  $200^\circ C$  at a rate of  $15^\circ C min^{-1}$ , and then to  $230^\circ C$  at a rate of  $100^\circ C min^{-1}$ . The carrier gas used was helium at  $23 mL min^{-1}$ . The injector and detector temperatures were set to  $200^\circ C$  and  $250^\circ C$ , respectively. Gas composition was quantified by gas chromatography (HewlettPackard 5890A) equipped with a thermal conductivity detector and two columns connected in parallel: Column 1, CP-Molsieve 5A ( $10 m \times 0.32 mm$ ) and Column 2, CP-PoraBOND Q ( $50 m \times 0.53 mm$ ). The gas carrier was argon at 3.5 bar pressure. The oven temperature was kept constant at  $40^\circ C$ .

### D. Biogas potential of raw manure

Complementary tests of biogas potential were performed in 3 parallels of 100 ml medical syringes. The syringes were filled with 30 ml raw manure with  $57 g VS L^{-1}$  and stored at  $20-23^\circ C$  in 169 days. Readings of the produced biogas was done frequently and the syringes were emptied regularly. The methane composition of the produced biogas was measured on day 64 when the biogas production was high.

### E. Calculations of AD process methane yield from raw manure

The overall process methane yield from raw manure is calculated in the following equation:

$$Y_{process} = \frac{Y_{AD} \times f_{separator}}{VS_{raw\ manure}} \quad (1)$$

where  $Y_{process}$  is the overall process methane yield ( $L$  methane  $g^{-1}$  VS in raw manure),  $Y_{AD}$  is the methane yield in the AD reactor ( $L$  methane  $L^{-1}$  feed),  $f_{separator}$  is the ratio between liquid in and out of the separator ( $L$  feed  $L^{-1}$  raw

manure) and  $VS_{raw\ manure}$  is the VS concentration in the raw manure ( $g VS L^{-1}$  raw manure).

## 3. Results and discussion

### A. Separation process

A 20-30 mm filter cake immediately established on the sieve mesh when used for the manure separation under vacuum. Mechanical filter cake removal was needed to maintain separation capacity since it clogged and caused high pressure drop. The raw dairy manure, containing 14 % added water for flushing, had a solid content of  $69 \pm 4 g TS L^{-1}$  ( $n = 2$ ) and  $56 \pm 4 g VS L^{-1}$  ( $n = 2$ ). Half of the TS content was removed during sieving (Table 2). The volume of the liquid fraction was 70 – 75 % of the raw manure volume sieved. The liquid manure  $COD_T$  content was  $62 \pm 12 g L^{-1}$  ( $n = 14$ ) and  $COD_S$  content was  $19 \pm 6 g L^{-1}$  ( $n = 11$ ) during the experiment, implying a large particulate COD fraction in the liquid manure after sieving.

Table 2. Liquid manure properties after sieving (based on n samples).

	Manure liquid fraction	n
TS ( $g L^{-1}$ )	$34 \pm 4$	6
VS ( $g L^{-1}$ )	$22 \pm 4$	6
$COD_T$ ( $g L^{-1}$ )	$62 \pm 12$	14
$COD_S$ ( $g L^{-1}$ )	$19 \pm 6$	11
$NH_4^+ - N$ ( $g L^{-1}$ )	$0.9 \pm 0.2$	3

### B. Biogas production rate and yield

The biogas production during the 5 months operation is shown in Figure 3. At  $25^\circ C$  the biogas production was around  $0.4 L$  biogas  $L^{-1}$  reactor  $d^{-1}$ . The temperature was increased from  $25 \pm 1^\circ C$  on day 41 to  $37 \pm 1^\circ C$  on day 60. The temperature was not stable in the reactor between day 41 and 60 due to technical problems with the heat control. The biogas production increased by 20 % to around  $0.5 L L^{-1}$  reactor  $d^{-1}$  due to the temperature increase. It then decreased at around 100 d when fresh manure was used. The highest biogas production rate measured during the study was  $0.76 L$  biogas  $L^{-1}$  reactor  $d^{-1}$  which corresponds to  $1.4 g$  COD biogas  $L^{-1}$  reactor  $d^{-1}$ .

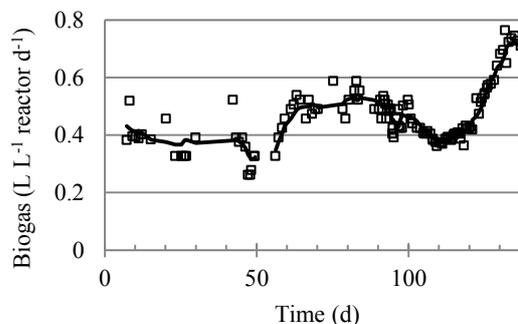


Fig.3. Measured biogas production rate and moving average (line).

Comparing the biogas production rate with the loading rate in Figure 4 it is clear that the methane production rate was much lower than the COD<sub>T</sub> loading rate. Particulates were evidently not broken down to a large extent in the reactor but TSS data (inlet 30 ± 4 g L<sup>-1</sup> vs outlet 26 ± 3 g L<sup>-1</sup>) suggest some biogas from particles. Methane production was also less than the COD<sub>S</sub> but higher than the COD<sub>VFA</sub> loading rate. This implies that more than just the VFA fraction of the COD<sub>S</sub> was converted to methane. Not measured VFA's may account for this difference.

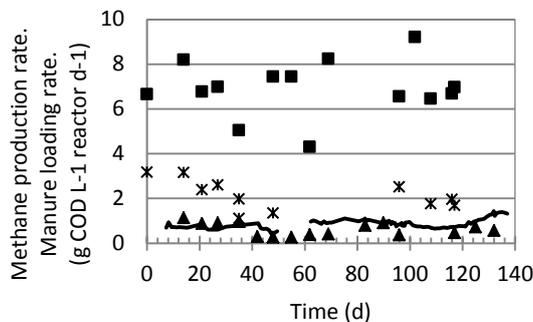


Fig. 4. Loading rate of COD<sub>T</sub> (■), COD<sub>S</sub> (\*) and COD<sub>VFA</sub> (▲)(g COD L<sup>-1</sup> reactor d<sup>-1</sup>) with moving average methane production rate (—) (g COD L<sup>-1</sup> reactor d<sup>-1</sup>).

The average yield at 37 °C from the whole study using stored manure (period B and E) was 157 L methane kg<sup>-1</sup> VS (Table 3) and the maximum yield achieved in the end of the test run was 220 L methane kg<sup>-1</sup> VS. Soluble components in the liquid fraction adding to the methane yield makes, however, a comparison based on VS incomplete [11]. A comparison based on COD, to take all the contributing components into account, shows 0.14 g COD methane g<sup>-1</sup> COD feed on average and a maximum of 0.19 g COD methane g<sup>-1</sup> COD feed. This is similar to the COD balance reported by Liao [12] testing biogas production from screened dairy manure at similar HRT at lab scale.

Table 3. Biogas yields and specific production rates at 37 °C.

	Biogas yield (L CH <sub>4</sub> kg <sup>-1</sup> VS)	Biogas yield (g COD biogas g <sup>-1</sup> COD <sub>T</sub> feed)	Biogas rate (L CH <sub>4</sub> L <sup>-1</sup> reactor d <sup>-1</sup> )	Biogas rate (g COD L <sup>-1</sup> reactor d <sup>-1</sup> )
Average for stored manure (Period B and E)	157	0.14	0.39	1.0
At maximum production.	220	0.19	0.54	1.4

### C. Biogas potential of raw manure

The biogas potential of the unsieved raw manure was 164 ± 7 L methane kg<sup>-1</sup> VS, based on a methane concentration

of 72 %. This is comparable to results of Møller [13] who got 148 ± 41 L methane kg<sup>-1</sup> VS (the value varied between 100 and 207) from 5 different farms with different feeding practice. A large fraction of the organic matter in the manure is not degradable by AD within a reasonable time frame, as can be seen from the change of VS content during the biogas potential test with 57 g VS L<sup>-1</sup> at the start and 47 g VS L<sup>-1</sup> at the end. Here 82 % of the VS content of the manure was not degraded implying that only 18% of the total organic matter of the manure was available for biogas production.

### D. Calculated methane yield for the overall process

The methane yield in the AD reactor was 3.6 L methane L<sup>-1</sup> feed based on the measured average production (period B and E), and the calculated AD process yield (Y<sub>process</sub>) was 47 L methane kg<sup>-1</sup> VS raw manure. Compared to the methane yield of the raw manure this implies that 29 % of the total methane potential of the raw manure was recovered by AD of the sieved fraction. A fraction of the total methane potential is also in the effluent as dissolved methane, VFA and organic particles (e.g. microorganisms), implying that less than 71 % remained in the solid fraction removed by the sieve. A methane production yield of 29 % is assumed to be an acceptable level if it can be produced much cheaper than the 100 % achievable in a process without solids separation with very long HRT and if the farmer obtain added value from the removed solids as dry composted fertilizer.

### E. Effect of manure storage on biogas production

Gas production was lower when using the substrate in periods C and D, which had been stored for less than 30 days compared to the manure with longer storage time used in periods B and E (Figure 5).

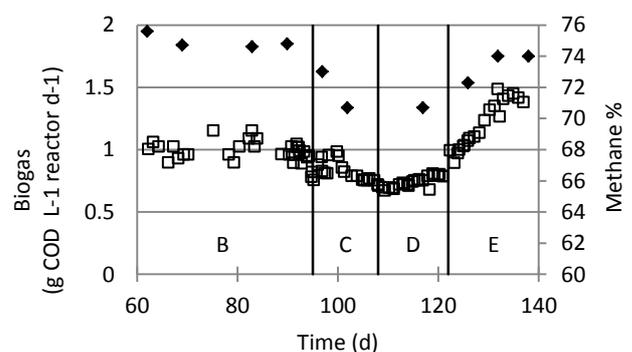


Fig. 5. Biogas methane content (◆) and biogas production rate (□) during periods B-E of different manure feed history. B; winter stored manure, C; stored 0-30 days, D; not stored and E; winter stored manure.

The substrate used in period D that had not been previously stored, gave the lowest biogas production rate of all, however, slowly increasing as the feed is maturing in the feed tank (Figure 1). The average methane content in the biogas during period B was 74.9 % ± 0.5 %. A reduction in the methane concentration to ~70 % coincides with the observed reduction in the biogas production (Figure 5). This indicates that storage affects both the biogas production rate and the methane concentration. This shift also influences the VFA of the

substrate and effluent (Table 4). The detected volatile acids are higher in stored than in fresh manure while there was less differences in effluent VFA. This can be explained by disintegration and hydrolysis of particulates to more easily degradable organic material during storage.

Table 4. VFA components in liquid manure feed and reactor effluent during change of raw manure storage source.

	Period	Acetic acid	Propionic acid	Butyric acid (iso+n)
Feed (g L <sup>-1</sup> )	B	3.9	1.2	0.5
	C	1.9	0.4	0.2
	D	2.3	0.5	0.3
	E	3.2	1.0	0.4
Effluent (g L <sup>-1</sup> )	B	0.6	0.1	
	C	0.4		
	D	0.8	0.1	
	E	0.9	0.2	0.02

#### F. pH and alkalinity

The pH of the feed ( $7.7 \pm 0.17$ ;  $n = 17$ ) is generally lower than the effluent ( $7.9 \pm 0.15$ ;  $n = 17$ ) of the reactor (Figure 6). VFA removal is assumed to be the main cause of the pH increase. The lower feed pH of manure used in periods A and B, that included manure stored during winter (ambient temperatures below 0 °C), can be explained by higher VFA content after long storage times.

The substrate total alkalinity level is 8-9 g CaCO<sub>3</sub> L<sup>-1</sup> ( $n = 3$ ). The relatively low amount of easily degradable organic components (Figure 4) and high alkalinity levels implies that process failure due to low pH caused by feed overload is not likely to occur. The HRT can, therefore, be reduced significantly to obtain much higher biogas productions rates than tested here without risking a detrimental pH drop due to accumulation of fatty acids in the reactor.

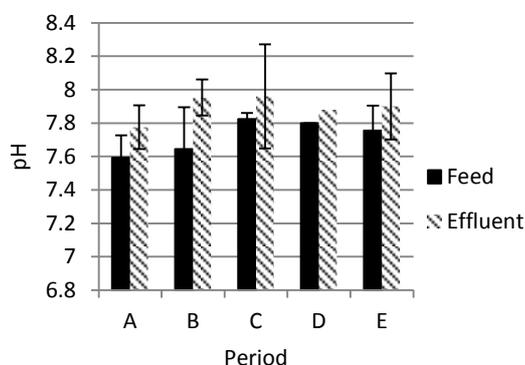


Fig. 6. pH of feed and effluent (days 30-60 not included in period A).

#### G. Sludge conditions in the pilot reactor

At start-up the influent TSS ( $29 \pm 4$  g L<sup>-1</sup>) was twice as high as the effluent TSS, explained by accumulation of solids. This difference was reduced during the first weeks

of operation until a difference of 3 g L<sup>-1</sup> was reached after 21 days and remained at that level for the duration of the test. This accumulation of solids in the reactor is assumed to have contributed to the development of a sludge blanket. This is a characteristic feature of a high rate reactor to obtain HRT  $\ll$  SRT (sludge retention time) to avoid sludge wash out at low HRT. The microorganisms must be trapped in a suspended biofilm or granular sludge for this purpose. The feed TSS content applied was, however, much higher than the upper level of 6 g L<sup>-1</sup> considered good conditions for granular sludge development [14]. This suggests that the sludge blanket consisted of some suspended biofilm form different from granular sludge. Visual inspection of samples from the sludge blanket support this as it consisted of particles of varying sizes (not quantified) but no typical granules were seen.

#### H. Practical solutions and further plans.

The results show that integrating an AD process with typical Norwegian farm infrastructure as proposed here is feasible. The installation and operation of such need to be well planned to obtain the observed benefits of manure storage prior to AD treatment.

The HRT was kept constant at 8.8 days during the whole experiment to limit the numbers of variables. Lower HRT may be required to allow for a smaller reactor size to gain the full benefit of the proposed process. Effects of reducing the HRT will, therefore, be tested at a later stage to test process limitations to avoid risking detrimental wash out of the microorganisms.

It is also desirable to gain more insight to the separation process to: 1. Obtain a liquid fraction with fewer particles for AD treatment. 2. Test different separation techniques that may be suitable for different types of manure. Manures which separate easily into solid and liquid phases by gravity (e.g. pig manure) may be cheaper to prepare and may contain fewer particles after extraction of the liquid fraction, and should also be examined as substrate for a HRAD process.

## 4. Conclusion

High rate AD of dairy manure can be maintained in a sludge blanket reactor after removing half of the TS content by a sieve. The maximum methane yield was 0.19 g COD biogas g<sup>-1</sup> COD sieved manure and the methane production rate was 0.7-1.5 g COD L<sup>-1</sup> reactor d<sup>-1</sup> at 8.8 days HRT. The gas production rate and yield increased with raw manure storage time. High manure alkalinity ensures stable pH at high load. The overall AD process gained 29 % of the total methane potential of the raw dairy manure and can give sustainable manure treatment.

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