## Methane Production in a 100-L Upflow Bioreactor by Anaerobic Digestion of Farm Waste

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#### **Abstract**

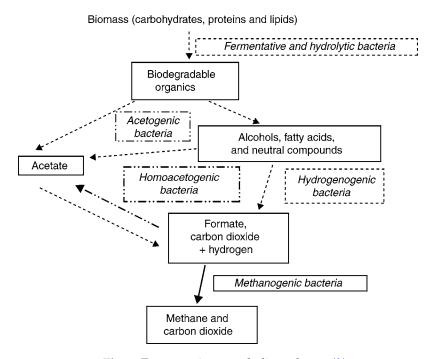
Manure waste from dairy farms has been used for methane production for decades, however, problems such as digester failure are routine. The problem has been investigated in small scale (1–2 L) digesters in the laboratory; however, very little scale-up to intermediate scales are available. We report production of methane in a 100-L digester and the results of an investigation into the effect of partial mixing induced by gas upflow/recirculation in the digester. The digester was operated for a period of about 70 d (with 16-d hydraulic retention time) with and without the mixing induced by gas recirculation through an internal draft tube. The results show a clear effect of mixing on digester operation. Without any mixing, the digester performance deteriorated within 30–50 d, whereas with mixing continuous production of methane was observed. This study demonstrates the importance of mixing and its critical role in design of large scale anaerobic digesters.

**Index Entries:** Anaerobic digestion; animal manure; gas recirculation; mixing; biogas.

#### Introduction

Methane produced by animal wastes is a clean replacement for coal and other fossil fuels which negatively affect air quality. Animal wastes represent a large unused source of sustainable, affordable, and renewable energy. In the United States, at least one billion tons of animal wastes are generated annually which is equivalent to approx 100 Mt coal/yr (1).

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**Fig. 1.** Fermentation metabolic pathway (3).

Methane is also a cleaner energy than traditional fossil fuels since it is compliant with policies of the Clean Air and Energy Policy Act representing a fuel source that can reduce  $\mathrm{SO}_{\mathrm{X}}$  emissions (biomass contains low amounts of sulfur), reduce  $\mathrm{NO}_{\mathrm{X}}$  emissions (biomass contains less nitrogen than coal), and reduce methane (formed in degradation of unused biomass) released into the atmosphere (2). The complex organics in animal wastes produces methane through a process that includes four main microbial cultures which work together to break down the waste, producing fatty acids that are further broken down to produce methane. The fermentation process (Fig. 1) consists of two final steps in which 70% of the methane produced is metabolized from acetate and 30% from carbon dioxide reduction with hydrogen (3).

Anaerobic digesters can be used to effectively process these wastes and collect the released methane; however, the effect of hydrodynamics and mixing in anaerobic digesters are not well studied. This could possibly contribute to the high-failure rate encountered in digester applications. The Department of Energy recently reviewed the history and performance of large anaerobic digesters implemented by the farming community for the treatment of animal wastes and the generation of methane for energy production (4). A total of 94 digesters of various designs were investigated as part of the study. The designs were categorized as

1. Plug-flow digesters—These digesters are of simple design in form of a trough, and a slurry mixture is fed once a day to one end of the

digester. The dimensions are in the range of 1:5 (channel width to length), and the total size is determined by the size of the daily feed. An expandable cover is used to collect the biogas. The hydraulic retention time (HRT) is on the order of 20–30 d, and the solids concentration is 11–13%. The plug-flow digesters are sensitive to the amount of solids present in the feed, since the feeding of the solids to one end, provides the "pushing action" to drive the content towards the other end.

- 2. Complete-mix digesters—These digesters have internal mixing and are usually similar to a chemical reactor—tall, circular, heated, with good controls. They suffer from high capital and maintenance costs. Sizes range from 95,000–1,900,000 L. The concentration of the solids are 3–10% and the HRT is 10–20 d.
- 3. Slurry digesters—The slurry digester operates in the same solids regime as the plug flow and complete-mix digesters. They require no mechanical mixing and are often constructed in silo configurations where internal convection (from temperature gradients and gas evolution) provides mixing.
- 4. Covered lagoon digesters—The lagoon digesters are used to treat streams with low solids concentration (<3%). It is a popular method used for methane production, in which the manure cleaning is accomplished by a flushing mechanism, generating large volumes of low-solid waste. The HRT is on the order of 60 d, implying that the conversion rate is very slow. It can take a couple of years to reach steady-state conditions in the lagoon. A floating cover collects the methane. The cost of these digesters is low and they are not heated, making methane production very dependent on the weather conditions.
- 5. Miscellaneous—Other types of digesters include designs that are not yet commercialized for farm use, such as upflow sludge blanket reactors and sequencing batch reactors. These types have potential for faster processing rates, but rely on more complex designs and thus higher capital and operational costs.

Of the 94 digesters reviewed, only 74 had actually been constructed, whereas the others were still in the planning stages or were never built. Only 28 of the digesters were in operation, the others had been shut down, either as a result of operational difficulties of the digester or by termination of farm operations. Based on the data available, it was determined that the failure rate was 50% overall. In the case of plug-flow and complete-mix digesters, the failure rate was 63% and 70%, respectively. No failures were seen with the slurry digesters, but the report concluded that there were too few cases (seven in total), to get an accurate estimate of the failure rate. The majority of failures were attributed to poor designs and installation, improper equipment and incompatible choice of materials of construction, and incorrect operation and lack of maintenance. The conclusion

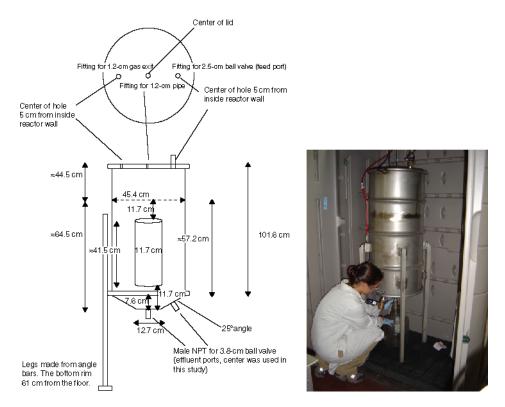


Fig. 2. Picture of the digester and collection of effluent.

of a poor design is usually indicative of inadequate mixing, resulting in plugging problems, problem obtaining desired pH balance, insufficient gas production, sand build-up, and so on. Improper choice of equipment and wrong choice of materials resulted in mechanical failures and in corrosion of materials from sulfur gases.

Thorough mixing of the substrate in the digester is regarded as essential in high-rate anaerobic digesters (5,6). The importance of mixing in achieving efficient substrate conversion has been reported by several other researchers (7–9), but the optimum mixing pattern is a subject of much debate. Some reports indicate that an intermediate degree of mixing appear to be optimal for substrate conversion (10).

In one of our previous studies (10), experiments with small-scale (4 L) digesters mixed via gas recirculation showed that the performance was independent of mixing rate and that unmixed digesters performed equally well. The purpose of the current investigation was to conduct larger-scale digester experiments and compare their performance to small-scale digesters.

#### Materials and Methods

The pilot-scale, stainless-steel digester held approx 96 L of bovine waste with 80 L of head space (Fig. 2). The digester was operated with a

16 d hydraulic retention time (HRT) and an average manure feed rate of 700 g total solids (TS) per day (containing approx 310 g total volatile solids [TVS]/d). These conditions were selected based on results from previous small-scale experiments conducted under different mixing conditions, amounts of TVS loaded, and hydraulic retention times (data not shown).

The digester was housed in a temperature-controlled (35°C) area (see Fig. 2). In order to provide mixing in the digester, gas was pumped from the top of the reactor by gas pumps and returned, from the top, to the digester at the lowest point of the draft tube insert. The cylindrical draft tube causes the gas to be directed through its interior which in turn causes the liquid to rise and mix. The gas recirculation rate through the digesters was approx 6 L/min. The digester was also operated under nonmixed conditions. In the mixed condition, the gas from the headspace of the digester was recirculated continuously through the digester for a period of 73 d. In the non-mixed condition, immediately following the previous phase, the digester was operated for a period of 66 d. On the 67th day, the mixing was reinitiated by starting the gas flow but other parameters remained unchanged. The digester was then operated in the mixed condition for an additional 16 d. The gas generated in the digesters was collected in Tedlar gas bags, and when these were full, the flow passed through an oil-filled wet gas meter (GSA/Precision Scientific, Chicago, II) capable of determining cumulative gas volume produced. This allowed measurement of total gas generation between sampling events.

The digester was operated using bovine manure collected from a dairy farm in the Anderson County, TN area. The cow manure was obtained fresh (i.e., <7-d old) from grass-fed cows kept in a pasture under no antibiotic treatment (i.e., antibiotic treatment of cows limits the viability of methane generating microorganisms in the cow manure). The cow manure was then refrigerated at 4°C until use. Before feeding the digester, the manure was prepared by blending tap water and wet manure in a 1:3 ratio for 2 min with an impeller mixer and placed into a large bucket for the heavy solids (sand, and so on) to settle out. TVS were determined and the slurry was diluted as needed to an estimated 6.6% (w/v) and passed through a sieve with a 9.5-mm pore size.

Feeding events for the digester occurred every other day in which gas composition and cumulative gas production volume was determined. On the sampling day, 12 L of reactor content (effluent) was removed (see Fig. 2), and 10 L of feed and 2 L of tap water were then added to the top of the digester (see Fig. 3). Triplicate samples were collected from the feed and effluent for TS and TVS determinations. Samples were also collected for pH, total fatty acids, and alkalinity measurements after each feeding event. TS were determined by drying a known weight of slurry at 105°C overnight, and TVS by volatilization of a known weight of dried slurry at 540°C for a minimum of 45 min (11). Total alkalinity was assessed by titrating

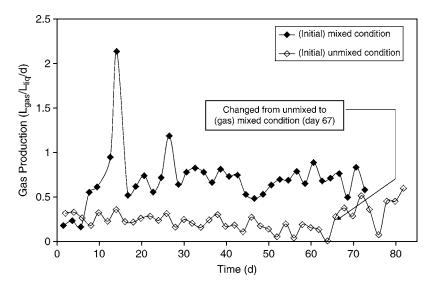


Fig. 3. Biogas productivity of the pilot-scale digester during different mixing conditions.

a known weight of manure slurry sample (10–20 g) with  $0.1\,M$  hydrochloric acid to a pH of  $4.5\,(12)$ .

Total fatty acids (TFA) analyses in the effluent and feed samples were performed by centrifugation and filtering samples through a 0.2-µm-pore-size filter, followed by injection of the filtrate into an HPLC. The mobile phase (filtered 5 mM  $\rm H_2SO_4$ ) of the HPLC was pumped at 0.6 mL/min through a 300 mm  $\times$  7.8 mm (8-µm-particle-size) RHM Monosaccharide column (Phenomenex, Torrance, CA) held at a temperature of 65°C to a refractive index detector (Model 2410, Waters Corporation, Miltford, MA) held at a temperature of 40°C. The sample injection volume was 10 µL and the resulting chromatograms were compared with injections of standards for acid concentration.

Gas samples (150 µL) were collected using a gas-tight syringe from a sampling port in the gas production line. They were injected in duplicate into a Hewlett Packard (Model 5890 Series II, Avondale, PA) gas chromatograph (GC) with a 30 m  $\times$  0.53 mm GS-Q phase capillary column (J&W Scientific, Folsom, CA). The injector, oven, and thermal conductivity detector (TCD) temperatures were 125, 50, and 150°C, respectively. The carrier gas (helium) flow rate through the column was 4 mL/min. The sample was injected in a split mode with approx 10% of the sample going through the column. The column make-up gas and the reference gas in the GC was helium. GC calibration of methane (CH4), carbon dioxide (CO2), and air gases was initially performed by injecting different volumes (50–200 µL) of pure gases. Later, periodic calibration was performed by injecting different amounts of air and using the relationship between TCD response factors (13).

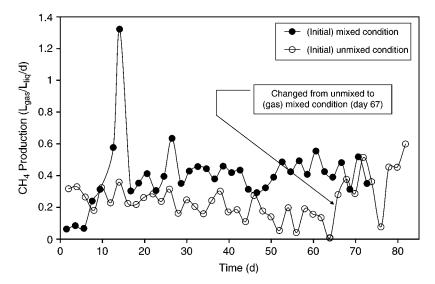


Fig. 4. Methane productivity of the pilot-scale digester during different mixing conditions.

#### **Results and Discussion**

The biogas (i.e., methane and carbon dioxide) and methane generation are shown in Figs. 3 and 4 for both the mixed (via recirculating gas) and unmixed operating conditions. It should be noted that the digester was operated continuously: 73 d mixed, 66 d unmixed, and 16 d mixed. Larger fluctuations were noted during the initial start-up phase, but the performance stabilized at approx 0.64 L biogas (or 0.4 L CH<sub>4</sub>)/L digester slurry volume/d when the digester was mixed. After an initial phase of predominantly  $\rm CO_2$  production (probably owing to presence of residual oxygen), the  $\rm CH_4$  content of the biogas was approx 60%  $\rm CH_4$  and 40%  $\rm CO_2$ , respectively (see Fig. 5).

In comparison with the mixed condition, the performance of the digester under the nonmixed condition was very different. The gas productivity of  $0.64~L_{\rm gas}/L_{\rm liq}/d$  observed under the mixed condition fell to more than half and, on certain days, there was negligible gas production under the nonmixed condition after 50 d of operation (Fig. 3). The methane production rate also decreased accordingly (Fig. 4) and the composition of the biogas became leaner (Fig. 5).

In order to confirm the effect of mixing, the gas recirculation was resumed on d 67. As observed from Figs. 3–5, the methane and biogas production picked up immediately upon starting the gas recirculation (indicated by an arrow).

The average amount of TS in the feed and effluent under the mixed condition at steady state were 115 and 83 g/L, respectively (*see* Table 1). At steady state the average amount of TVS in the feed and effluent were

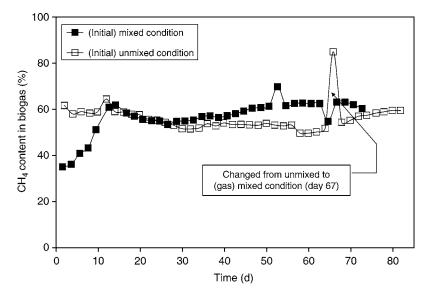


Fig. 5. Gas composition of biogas from the pilot-scale digester during different mixing conditions.

Table 1
Different Measured Parameters for Feed and Effluent at Steady State

	TS	TVS	TFA	$\mathrm{CH_4}$ productivity $(\mathrm{L_{gas}/L_{liq}/d})$
Mixed condition Feed <sup>a</sup>	115	52	5.5	0.39
Effluent	83	44	0.1	
Unmixed condition Feed <sup>a</sup>	98	47	3.1	0.14
Effluent	113	53	5.5	0.14

<sup>&</sup>lt;sup>a</sup>The feed concentration has been corrected for dilution.

52 and 44 g/L (Table 1), while the TFA of the feed and effluent at steady state were 5.5 and 0.1 g/L (Table 1), respectively. During start up, there was an excess of propionic acid in the effluent (data not shown), but upon achieving steady state conditions there was little acid found in the effluent.

After the digester had been operated in an unmixed mode for 60–66 d, the TS in the feed and effluent were 98 and 113 g/L (see Table 1) and the average concentration of total volatile solids in the feed and effluent were 47 and 53 g/L, respectively (Table 1). The apparently higher concentration of TS and TVS in the effluent demonstrates the difficulty of obtaining accurate operating conditions for unmixed conditions. It is likely that significant settling occurs during unmixed operation. Consequently, effluent samples

drawn from the bottom of the digester will not necessarily be indicative of the average composition of the digester. The incomplete conversion of TVS during unmixed operation corresponds to the poor methane productivity observed (Fig. 4). The TFA were higher in the effluent than in the feed (i.e., 5.5 vs 3.1 g/L). This suggests that the acetogenic bacteria are active as the fatty acids accumulated in (at least the bottom of) the digester, while the methanogenic bacteria were not. The alkalinity of the feed and effluent were essentially constant for both mixing conditions (data not shown).

The results obtained in this study contradicts the results obtained in our small-scale studies, where performance were unaffected by mixing conditions (10). In both studies, biogas recirculation were used as the mode of mixing, and it has been reported that biogas recirculation is the most efficient mode of agitation for anaerobic digesters (8,14,15). Although our current studies shows better performance under mixed conditions, higher methane production rates in unmixed digesters have been shown by Ghaly and Ben-Hassan (16); however, it has also been suggested that unmixed reactors perform worse, especially large reactors (17). It is possible that the size of digester is an important factor to consider, Ghaly and Ben-Hassan (16) found in their literature review that the digester units should have a diameter greater than 25 cm and a liquid depth of 20 cm, or greater, in order to provide reliable data that can be used for scale-up. The digester used in our pilot study fits this criterion, while the digesters used in our previous experiment did not.

#### **Conclusions**

Biogas production in a 100-L pilot-scale digester was evaluated and the effect of partial mixing induced by gas upflow/recirculation in the digester was studied. The digester was operated for a period of about 70 d (at a 16-d hydraulic retention time) with and without the mixing induced by gas upflow. A steady-state methane production of 0.4 L<sub>gas</sub>/L<sub>liq</sub>/d was obtained. This result is slightly lower than that obtained in the small-scale study (10). However, in the pilot-scale study, the results show a dramatic effect of mixing on digester operation. Without any mixing, the digester performance deteriorates within 30–50 d, while with mixing, continuous and consistent production of methane is observed. This study demonstrates the importance of mixing and its critical role in design of large-scale anaerobic digesters. Future work on this project is targeted towards application to larger-scale designs as well as studies with small-scale digesters to determine the effect of scale-up. Further areas of interest will be in optimizing the mixing parameters and comparison with mechanical mixing.

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