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Article

Performance of a Small-Scale, Variable Temperature Fixed Dome Digester in a Temperate Climate

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Abstract: Small-scale digesters, similar to popular Chinese designs, have the potential to address the energy needs of smaller dairy farmers in temperate U.S. climates. To assess this potential, a 1.14 m^3 (300 gallon) modified fixed-dome digester was installed and operated, at variable temperatures (5.3 to 27.9 °C) typical of the Midwestern United States, from March 2010 to March 2011 (363 days). Temperature, gas production, and other variables were recorded. The system was fed with dilute dairy manure with 6% volatile solids (VS) and an organic loading rate (OLR) ranging from 0.83 to 2.43 kg volatile solids (VS)/m^3/day. The system was loaded with no interruption and exhibited no signs of inhibition from July 2010 to mid-November 2010 (129 days). During this period the digester temperature was over 20 °C with an average daily biogas production of 842 ± 69 L/day, a methane yield of 0.168 m^3/kg VS added, and a Volatile Solids reduction of 36%. After the temperature dropped below 20 °C, the digester showed signs of inhibition and soured. These findings suggest that an ambient temperature, modified fixed dome digester could operate without temperature inhibition for approximately six months (169 days) a year in a temperate climate when digester temperatures exceed 20 °C. However, during colder months the digester temperature must maintained above 20 °C for viable gas production year round.

Keywords: anaerobic digestion; dairy manure; biogas; organic loading rate

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1. Introduction

In the U.S., where approximately 230 million tons of animal dry matter residues are generated every year [1], conventional digesters have been mainly developed for larger agricultural and industrial applications. These conventional digesters are mixed and heated to maintain operation at mesophilic (35–42 °C) or thermophilic (45–60 °C) temperatures to enable greater gas production [2]. Because these systems are mechanically complex and require specialized controls to operate successfully, their cost is usually greater than $500,000 [3]. Further improvements of conventional digesters have added more operational complexity such as dividing the anaerobic digestion process into two sequential stages in which a given feedstock is first converted into acids (acidogenic stage) followed by the final conversion of acids into methane (methanogenic stage) [4,5].

Due to the expense and scale of the conventional digesters, in 2011, 120 of a total of 143 dairy farm digesters were located at commercial facilities with herds containing more than 500 cows [6]. Although large-scale conventional digesters are established technologies for anaerobic digestion of animal and agricultural wastes for gas production, their affordability and application is limited for medium and small dairy farms. Operations with less than 500 dairy cows represent almost 95% of U.S. dairy farms. The absence of small-scale digester designs prohibits more than 60,000 medium and small dairy farmers from realizing the benefits of anaerobic digestion [7]. Therefore, there is a clear need for small-scale digesters and a large potential market of medium and small-scale dairy farms in the U.S.

In contrast to the situation in the United States, countries like China and India successfully use millions of small and medium scale digesters [8,9]. In these countries, biogas programs were promoted as a method to meet basic energy needs of rural farm families. In addition, several commercial pre-fabricated small-scale digester designs have been developed for applications in these countries. Such small and medium digesters are simple and low cost systems that require no mixing and heating for operation, but they require water to dilute the manure as slurry for optimal operation. These systems have been widely utilized in tropical and subtropical areas where ambient temperatures do not limit gas production. To date, more than 30 million small-scale digesters are in operation [10]. By 2006 in China, for example, 22 million of 250 million rural farmsteads had been equipped with small and medium scale biogas digesters [11]. Among existing small-scale digester designs, the Chinese fixed-dome digester is the most common and successful design and is a typical component of integrated small farming operations in China [12]. Adapting the small-scale digester designs that are successfully used in other countries may have potential to meet the need for small-scale digesters in the U.S. This could enable small and mid-size livestock farmers to realize the benefits of anaerobic digestion and improve their economic efficiency and environmental sustainability.

While small-scale digesters have been widely installed and operated successfully in tropical and sub-tropical areas [13], the low winter temperatures in temperate climates may limit their performance. Under low temperatures, due to the accumulation of volatile fatty acids (VFAs) [14,15], the digesters can become acidic with limited biogas production. However, previous studies have showed applications of anaerobic digestion at low temperatures. For example, the anaerobic digestion of liquid substrates with low solids concentrations at low temperatures has been completed with conventional wastewater reactors (Upflow Anaerobic Sludge Blanket and Expanded Granular Sludge Bed) arranged in two stages. In these reactors, the separation of phases (acidogenic and methanogenic) and a better contact between the substrate and microorganisms counteract the negative effects of low temperature [14,16–19]. Another study recommended the use of heated water for dilution in small-scale digesters to increase and maintain higher temperatures during the winter [20]. This study concluded that if the local average ambient temperature during the winter is above −5 °C, reliable year-round biogas production is possible. The year-round operation of small buried digesters in northern China and northern India where temperatures drop below 20 °C during the winter also signals potential success of these devices in temperate climates [8,9]. Hill *et al.* [21], working with very dilute dairy manure in low temperature lagoon digesters, reported the need for low loading rates of 0.1 and 0.2 kg VS/m^3/day during colder temperatures to achieve methane yields comparable to those obtained at mesophilic temperatures. Prior work has described the feasibility of using small-scale anaerobic digesters to produce biogas year-round in cold climates and the impacts of varying the organic loading rate [8,20–22]. However, these studies have either been theoretical with no experimental data, or have not focused on dairy cow manure as the feedstock.

Simple and affordable small-scale digesters, such as common in China, may have potential to fulfill the need for similar systems by medium and small dairy farmers in the United States. With these digesters medium and small dairy farmers can use biogas for heating or cooking rather than electrical generation as typical in larger dairy farms. We conducted this experiment to test the potential of adapting fixed-dome digesters to small dairies in temperate climates in the U.S. For this purpose a 1.14 m^3 buried digester, housed in a greenhouse to moderate temperature during cold months, was operated at ambient temperature and fed with dilute dairy cow manure (~6% volatile solids) so the effects of organic loading rates and temperature on biogas and methane production could be assessed for one year. The results were evaluated and compared with other small-scale and larger digesters.

2. Results

2.1. Temperature and Biogas Production

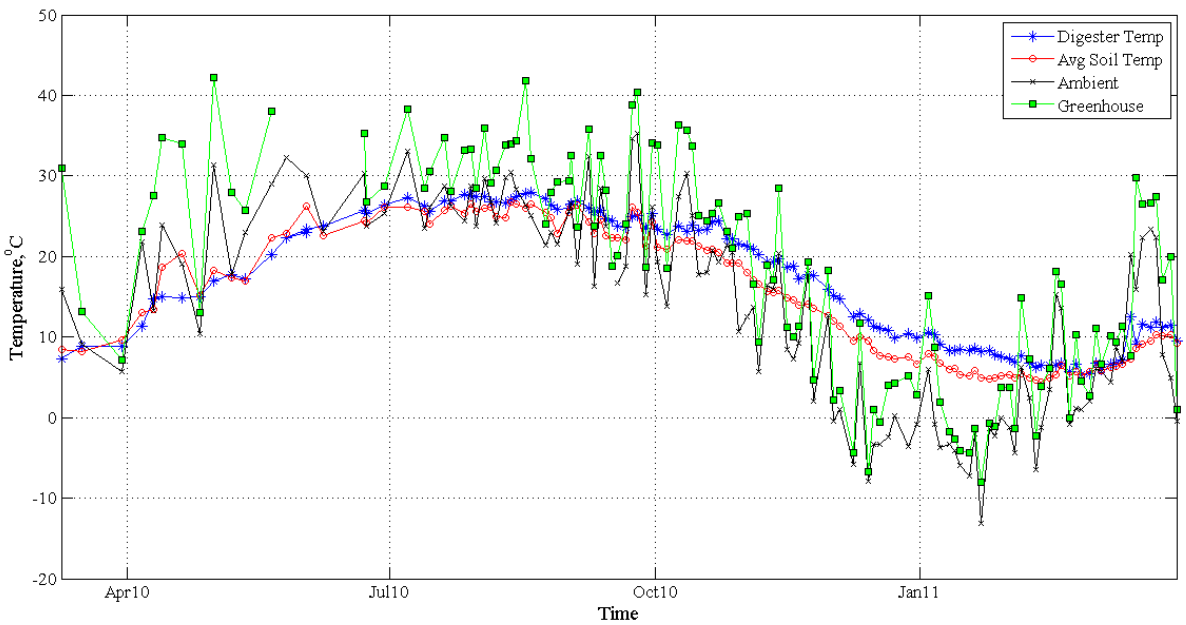
Insulating and burying the digester successfully increased the digester temperature above the ambient winter temperatures. Soil and digester temperatures were characterized by smaller year-round temperature ranges compared to the greenhouse and ambient temperatures (Figure 1). The ambient, greenhouse and digester temperatures ranged between −13.5 to 35 °C, −8.1 to 42.2 °C and 5.3 to 27.9 °C respectively. The greenhouse increased the average temperature by 4.4 °C above the ambient temperature, and the difference between the minimal ambient winter temperature and minimal digester temperature was 19.1 °C. Comparing the soil temperatures with ambient temperature, specifically during the winter, it was obvious that soil temperatures present lower variability with a mean of 6.1 °C (SD = 1.2) that is greater than the average ambient temperature 2.7 °C (SD = 7.8); hence, the digester temperature presented lower variability (Figure 1). The correlation analysis shows that the temperature of the slurry in the digester was highly correlated to average soil temperature (R^2 = 0.956), with the highest temperature in the digester (27.7 °C) recorded in the month of July (2010) and the lowest (5.9 °C) in the month of March (2011).

During the start up the average biogas production was 18.66 ± 8.6 L/day and the average methane concentration was 24.6% ± 0.08%. As a consequence, the loading was stopped for approximately

2 months (Initial Rest), and the digester was reseeded on May 11th (May–June 2010). The loading resumed 50 days later, once both biogas production and methane concentration increased over 800 L/day and 45% respectively. Although a larger start up time is desirable, the startup period of 50 days was sufficient and greater than other digester studies [23,24]. Afterwards, the digester exhibited more stable conditions and increased production of biogas and methane (Optimal Operation). The VFA concentration of the digester ranged from 1776 to 3162 mg HAc_{eq}/L, which was greater than recommended levels <700 HAc_{eq}/L [25]. Withstanding this fact, several factors demonstrated the digester had stable gas production and was operating efficiently due to a high buffer capacity that maintained the pH above 7. A microbial analysis of this digester found a predominance of *Methanosaetacea* which is typically found in stable digesters [26].

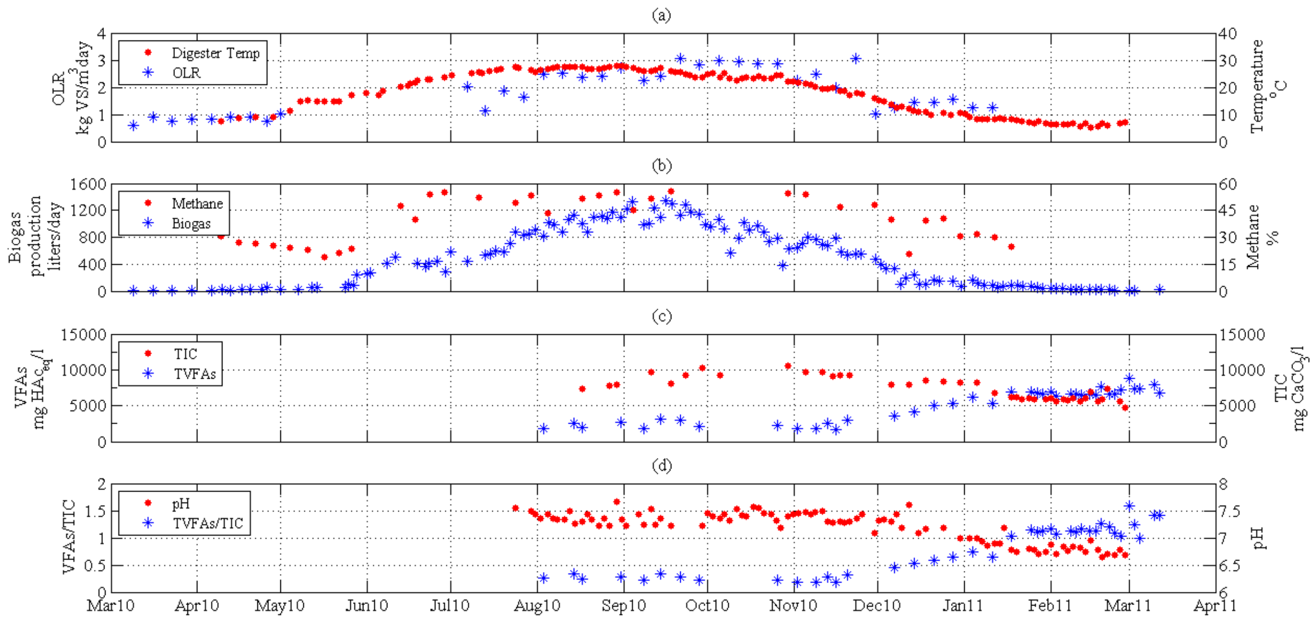
Also between July and November, the digester showed stability with the TVFAs/TIC ratio remaining far below 0.4 (Figure 2d), which indicates stable operation [27]. In addition, neither pH nor Alkalinity were below 7.3 and 8200 mg $CaCO_3$ mg/L respectively, and the mean TVFA concentration was 2222 mg/L. These conditions indicated that the digester did not exceed inhibitory thresholds between July and mid-November [27–29].

Figure 1. Temperature data for the digester, average soil, ambient and greenhouse from 8 March 2010–31 March 2011.



After this period of relative stability, the digester temperature dropped below 20 °C in mid-November (Figure 1), and the OLR was reduced to a value below 1.58 ± 0.4 kg VS/m^3/day in an attempt to prevent digester failure (Figure 2a). However, after the second week of November the pH decreased to 7.01 ± 0.08 as a result of an unbalance between TVFAs and TIC (Figure 2c,d). The TVFAs/TIC ratio rose from 0.25 to 0.48 in six weeks. By mid-January 2011, when the loading had been stopped, the TVFAs/TIC ratio was above 1.0 indicating that the digester was soured. Under these unfavorable conditions the average daily biogas production, methane concentration and methane yield decreased by 67%, 27% and 66% respectively. From mid-January to mid-March 2011, the digester was not loaded. At this final stage the average TVFAs concentration was 6917 ± 301 mg HAc_{eq}/L, and the average TVFAs/TIC ratio was greater than 1.0. These values confirmed that the digester was soured (Table 1) [27,28]. Even with the variation in OLR among the stages, a correlation analysis showed clearly that both biogas and methane production correlated with changes in the digester temperature (Pearson correlation coefficients r = 0.807 and r = 0.809, $p < 0.01$) (Figure 2b).

Figure 2. Digester operational conditions. (**a**) OLR and Temperature; (**b**) Biogas production and Methane Concentration; (**c**) Volatile Organic Fatty Acids (VFAs) and Total Inorganic Carbonate (TIC); (**d**) Volatile Organic Acids—Total Inorganic Carbonate ratio (VFAs/TIC) and pH. (Sampling period from 9 July 2010 until 3 November 2011 for (**c**) and (**d**). A broken instrument was the cause of the lack of data for VFAs between 29 September 2010 to 25 October 2010).



2.2. VS Reduction

The measure of VS reduction is an indirect measurement of organic matter utilization. The VS/TS ratio for the diluted manure feedstock and the digestate effluent were 0.83 and 0.78 respectively, which indicates that a fraction of the organic matter present in the diluted manure was transformed into methane and carbon dioxide. During a year of operation the mean VS concentration of the feedstock and digestate were 65,000 ± 3,000 and 41,000 ± 2,000 mg/L respectively. The variability in the feedstock is somewhat related to daily and seasonal variability in moisture content of the manure collected and the ratio varied accordingly. The VS concentration in the digestate was estimated to be 20,000 mg/L to 27,000 mg/L lower than the VS concentration in the feedstock (95% CI, two sided *p*-value = 0.005 from a two- sample *t*-test), which indicates the digester removed an average of 34.8% (SD = 10%) of the influent VS solids concentration. However, as is shown in Table 1, the efficiency decreased over time from an average of 40.1% (SD = 8%) during the start-up (March–April 2010) to an average of 25.6% (SD = 11%) (November 2010–January 2011) when the digester exhibited poor performance.

Table 1. Main operational variables during the five stages of digester operation. (95% confidence intervals, $\hat{x} \pm \theta$). Statistical differences of operational data among stages were conducted through unbalanced one-way Anova, and Tukey–Kramer multicomparison tests were used for comparisons among stages.

Parameter	**Five stages of operation** [d]				
	1. Start up	**2. Initial Rest**	**3. Optimal Operation**	**4. Poor Operation**	**5. Final Rest**
	8 March 2010–30 April 2010	1 May 2010–30 June 2010	1 July 2010–10 November 2010	11 November 2010–10 January 2011	11 January 2011–11 March 2011
OLR [a], kg VS/m^3/day	0.83 ± 0.08 [I]	0.00	2.43 ± 0.22 [II]	1.58 ± 0.4 [III]	0.00
Temperature, °C	12.7 ± 1.89 [I]	22.74 ± 1.37 [II]	25.04 ± 0.60 [III]	13.42 ± 1.44 [IV]	7.05 ± 0.38 [V]
Total Solids influent, mg/L	74,000 ± 8,000 [I]	–	81,000 ± 4,000 [I]	80,000 ± 6,000 [I]	–
Volatile Solids influent, mg/L	61,000 ± 6,000 [I]	–	67,000 ± 3,000 [II]	63,000 ± 5,000 [I,II]	–
Total Solids effluent, mg/L	42,000 ± 2,000 [I]	–	58,000 ± 4,000 [II]	60,000 ± 5,000 [II]	–
Volatile Solids effluent, mg/L	36,000 ± 1,700 [I]	–	44,000 ± 2,500 [II]	46,000 ± 2,000 [II]	–
Volatile Solids reduction, %	40.1 ± 5.1 [I]	–	35.8 ± 4.6 [I]	25.6 ± 6.7 [II]	–
Biogas, liters/day	18.66 ± 8.6 [I]	268 ± 86 [II]	914 ± 63 [III]	302 ± 91 [IV]	39 ± 11 [I]
Methane, %	24.6 ± 2.25 [I]	49.6 ± 5.5 [II]	51.55 ± 2.39 [II]	37.24 ± 6.30 [III]	27.43 ± 5.34 [I]
Methane Yield, m^3 of CH_4/kg VS added	0.005 ± 0.002	–	0.176 ± 0.005	0.06 ± 0.013	–
TVFAs [b], mg HAc/L	–	–	2222 ± 276 [I]	4074 ± 980 [II]	6917 ± 301 [III]
TIC [c], mg $CaCO_3$/L	–	–	8904 ± 652 [I]	8544 ± 350 [I]	6085 ± 272 [II]
TVFAs/TIC	–	–	0.25 ± 0.03 [I]	0.48 ± 0.13 [I]	1.15 ± 0.08 [II]
pH	–	–	7.39 ± 0.03 [I]	7.01 ± 0.08 [II]	6.81 ± 0.04 [III]

$\hat{x}$ —mean; $x \pm \theta$ —lower and upper endpoints of confidence interval; [a] OLR—Organic loading rate; [b] TVFAs—Total volatile fatty acids; [c] TIC—Total inorganic carbonate alkalinity; [I, II, III, IV] and [V] superscripts are used to show multiple comparisons of group means. Different superscripts between operational stages indicate marginal means are significantly different

3. Discussion

Unlike previous studies that address the performance of small-scale buried digesters in temperate regions, these results demonstrate how burial can moderate and increase the digester temperature in temperate regions during cold seasons. However, the average digester temperature during the stages of start-up and final rest was well below 20 °C, and too low to support acceptable rates of digestion and energy production. The ambient temperature variations observed in this study (Figure 1) are similar to temperature variations in other temperate countries such as Romania, Armenia and Kyrguizstan and Sichuan Province in Southern China where small-scale digesters have operated at least five months of the year during periods when temperatures were above 20 °C [8,20]. As opposed to another study that reported acceptable biogas production at digester temperatures as low as 15 °C with methane contents between 56%–58.5% [27], our results showed acceptable biogas production for temperatures above 20 °C. At these temperatures, occurring during the optimal operation, the digester showed a methane yield of 0.176 m^3 CH_4/kg VS added that compares well with previously reported methane yields at 24.5 °C [27] (0.180 m^3 CH_4/kg VS added) and 22.5 °C [28] (0.132 m^3 CH_4/kg VS added).

In contrast, the average biogas production decreased 77%, with respect to the optimal operational stage, when the digester operated at temperatures below 20 °C (Table 1). During this period of poor performance the average temperature in the digester was 13.42 °C, and a methane yield of 0.06 m^3 CH_4/kg VS was only 43% of that in a Deenbandhu digester during the coldest month [27]. In this study, digester temperatures below 20 °C resulted in unstable performance characterized by increased TVFAs concentrations (>3,000 mg/L HAc_{eq}). The amount of biogas produced during these temperatures is not sufficient to permit year-round use. Significant design and management improvements are needed to increase digester temperatures to permit year-round use of variable temperature digesters in temperate climates.

The VS reduction showed no dependence on temperature (Table 1) with average values that are similar to both small-scale and farm-scale digesters. After a year of operation, an average reduction of 34.8% was consistent with results obtained from other digesters fed with cow manure. A study of a Janata fixed dome digester in India, for example, reported a VS reduction of 32% during the warmest month of operation [28]. Another study reported an average VS reduction of 29.7% for a farm scale digester operated at a constant temperature of 35 °C [29].

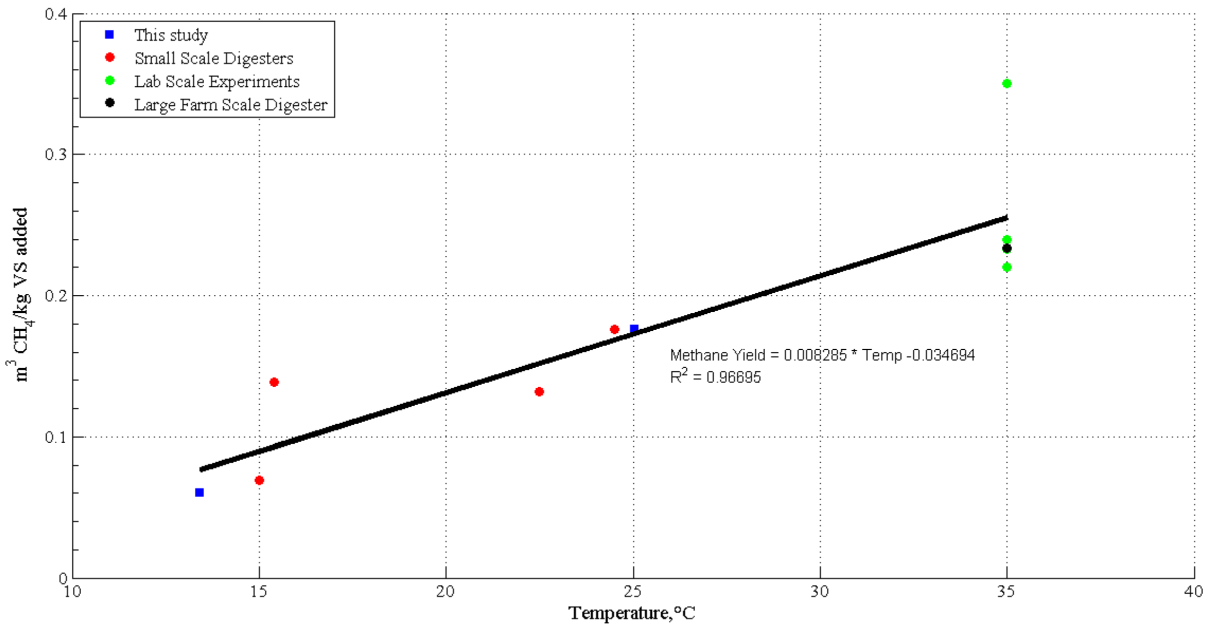
Despite the fact that average VS reduction was comparable to other digesters, the digester had decreased VS reduction during the year of operation from an initial 40.1% to a final 25.6% (Table 1). This occurred because digesters fed with cattle manure often accumulate a top layer of lighter undigested slurry that increases the amount of solids in the effluent. In addition, dairy manure has a high content of undigestible organic matter that settle within the digester and shorten the hydraulic retention times affecting the settling efficiency [30,31]. Both phenomena may explain increases of the VS concentration in the effluent from March 2010 to January 2011 (Table 1). Thus, for better digester operation, this undigestable material described above should be periodically removed, at least once a year, from the digester [8].

In this study we insulated the digestion chamber with polyurethane foam; however, the lack of insulation of the displacement tank, likely resulted in lower digester temperatures than a minimum of 15 °C below which several authors have reported substantial reductions in methane production rates [32]. This occurred as colder slurry in the displacement tank entered the digester several times a day when the gas pressure was released [9]. Although we made no measurements of the volume returned back to the digestion chamber, we estimate that it may have represented up to 10% of its useful volume. Future designs should include insulation of the digester and the loading and displacement tanks.

It is clear from our results that during the cold months the digester temperature was not sufficient to permit the current design to produce a viable amount of biogas. However, the results obtained from July to November 2010 (129 days), when the digester was fed with no interruption, and the digester temperature was above 20 °C compare favorably to previous results and demonstrate stable operation with no signs of inhibition due to increases in TVFAs or decreases in total inorganic carbonate alkalinity and pH [23,33]. Hence, if a stable operation and viable gas production occur at temperatures higher than 20 °C, a fixed-dome small-scale digester could operate approximately six months a year (169 days) in the Midwest of U.S. Comparison with the performance in terms of methane yields of previous digesters confirms this point (Figure 3). During the period of optimal performance (Table 1) the OLR used (2.43 kg VS/m^3/day) and the methane yield obtained (0.176 m^3 CH_4/kg VS added) were

47% and 28% greater than the values calculated from a Janata fixed dome digester ADII at 22.5 °C (1.3 kg VS/m^3/day, 0.1265 m^3 CH_4/kg VS added) [34]. Additional studies of small and medium-scale digesters have reported similar biogas yields and stable operation at temperatures above 20 °C [34–36]. Compared with a conventional digester, this OLR and the methane yield represent almost 87% and 75% of those values from a farm scale digester (550 dairy cows) at 35 °C [29]. Compared with controlled lab experiments at mesophilic and thermophilic temperatures, the methane yield obtained was 51% of the ultimate methane yield obtained from a set of batch digesters fed with cattle manure and maintained at 30–60 °C (at 5 °C intervals) [37]. In contrast, the biogas and methane yield decreased far lower when the digester slurry temperature was below 20 °C. At these temperatures the digester also showed signs of unstable performance with TVFAs concentrations over 2,000 mg HAc_{eq}/L and decreasing alkalinity and pH (Figure 2c).

Figure 3. Methane Yields of digesters fed with cow manure. (■) Data from this study; (●) Data from other small-scale digesters with no controlled temperature [27,38]; (●) Data from lab experiments where digesters had controlled temperature [30,34,37,39]; (●) Large farm digester with controlled temperature [29].



Based on recommendations from previous research [20], we tried to maintain the performance of the digester as the temperature decreased by reducing the OLR to target value of 1.5 kg VS/m^3/day [30]. For this purpose, we used warm water for dilution, and intentionally increased the mixing by recirculating digestate from the compensation tank to the loading tank.

During the warmer months of operation (July to November 2010) a greater OLR (2.4 kg VS/m^3/day) was maintained to yield a higher production of methane and biogas. In an attempt to maintain the digesters performance as temperature decreased a TVFAs/Total Inorganic Carbonate Alkalinity ratio below 0.4 was used as an indicator of stability [25]. This criterion is more conservative than the widely used value of 0.4 for TVFAs/Total Alkalinity ratio [30]. As this ratio exceeded this threshold in November we decreased the OLR from 2.4 to 1.5 kg VS/m^3/day. However, this change did not stabilize the digester. On the contrary, four weeks later the digester became soured with VFAs exceeding 4,000 mg/L and TVFAs/TIC ratio higher than 0.75 [30]. Maintaining the OLR of 2.4 kg VS/m^3/day into November was likely a mistake that lead to increased concentrations of TVFAs and digester failure when the temperature dropped below 20 °C. Better management guidance is needed for these digesters to maintain performance and avoid digester failure during decreasing temperatures. These results suggest that the threshold value of TVFAs/TIC ratio of 0.4 for an optimal operation is an unsuitable indicator of stability at mesophilic and lower temperatures [25]. Therefore, this threshold value should be decreased in order to indicate when to lower the OLR in digesters operated at variable and, especially decreasing temperatures. In addition, the knowledge of the concentrations of specific TVFAs, such propionate acid, or ratios of specific Volatile Fatty Acids, such as propionate/acetate, maybe better indicators of the stability of variable temperature digesters [31].

Previous studies with small-scale digesters suggest the use of constant and lower organic loading rates during the year for stable operation at lower temperatures. For example, it is stated that the OLR should not exceed 1.5 kg VS/m^3/day [8]. It was also concluded that digesters fed with OLR rates between 0.1 and 0.2 kg VS/m^3 exhibited stable operation [33]. However, an average OLR of 2.4 kg/m^{3}day during the warmer months resulted in a methane yield that is 39% higher than the performance of similar digesters [38]. Despite this apparent advantage of using a higher OLR during the warmer months, the results during the colder period showed signs of poor methane production (0.06 m^3 CH_4/kg/VS day) and signs of inhibition (TVAs > 4,000, decreasing pH).

Using a lower organic loading rate during the warmer months of operation may help avoid digester failure during the transition from warmer to colder months. Although our results demonstrate that biogas production is highly affected when the digester temperature drops below 20 °C, other studies showed an acceptable biogas production at digester temperatures as low as 15 °C, but with lower and constant OLR [27]. While this may result in lower gas and methane production during warmer temperatures, it may extend digester operation without risk of system failure when temperature drops to 15 °C; however, under this low OLR, the digester volume needed would be exceedingly large for a given farm application [9,22,38]. Other researchers have evaluated approaches to maintain higher digester temperatures, including supplemental heating to maintain the digester temperature above 20 °C during cold months [35,40]. Although in this approach a substrate other than cow manure was used, the biogas production was acceptable compared to that in mesophilic digesters. This approach could be explored by using a minimum temperature threshold of 20 °C, where the digester only would need to be heated for six months a year. Keeping the OLR as high as possible by heating the digester also results in digesters with smaller volume.

4. Experimental Section

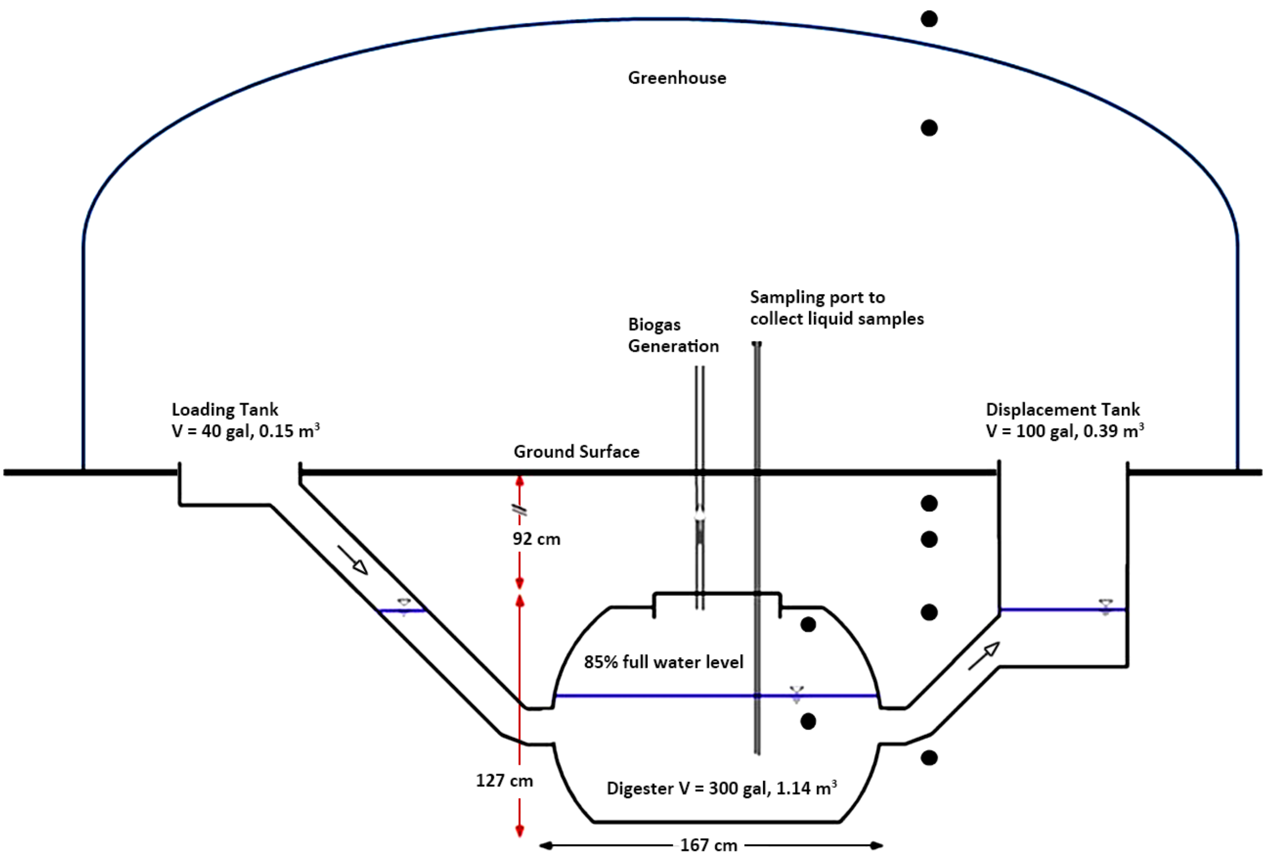
4.1. Study Site

This study was conducted at the Waterman Dairy Facility located on the campus of Ohio State University (40°00′34.1″ N, 83°02′31.3″ W). The milking herd consisted of 80 Jersey cows housed in free-stall barns. The diet through the year included a mixture of silage, alfalfa and grain with ratio of approximately 45:10:45.

4.2. Modified Fixed Domed Digester

A polyethylene tank with a thickness of 3.2 mm was used to create a modified Chinese fixed dome digester based on typical Chinese designs [20,41]. As with typical fixed dome digesters the design consists of three main components (Figure 4): (1) A 0.15 m^3 loading tank where fresh manure was diluted prior to loading the digester; (2) a 1.14 m^3 digestion chamber with 85% working volume occupied by slurry and 15% gas storage space; and (3) a 0.39 m^3 displacement tank. These components are connected by 15.24 cm PVC pipes (Figure 4). To insulate the digester it was sprayed with polyurethane foam (Tiger Foam™ insulation) to a thickness of 2.5 cm and buried inside a greenhouse to a depth 2.19 m. The greenhouse, in which the digester was located, was not insulated, heated, or completely sealed, but did protect the experimental equipment from rain and wind.

Figure 4. Modified fixed-dome digester used for this study. Black dots indicate the locations where thermocouples recorded temperature.



4.3. Digester—Instrumentation

The digester was instrumented to record temperature, gas pressure and gas production. Eight k type thermocouples with silicone isolated junctions were installed: four measured soil temperature at 15, 30, 80 and 150 cm below the soil surface, two ambient and greenhouse temperatures, and two temperatures inside the digester. A manometer (1223-12-W/M, Dwyer Instruments, Michigan City, IN, USA) recorded pressure in the gas line. In addition, a solenoid valve was installed on the gas line. This valve released the gas from the digester chamber once the pressure exceeded 18 cm of water pressure.

When the pressure fell below 2.5 cm of water pressure the valve closed to start building pressure again. This automated pressure control promoted more internal mixing by displacing the digester chamber slurry through the inlet and outlet pipes connected to the loading and compensation tank [9]. To record gas production, a gas meter (Zhejiang Songchuan Meter Technology Stocks Co., Ltd., Taizhou, Zhejiang, China, Ref.: JBD2.5-SA), with a precision of 0.1 L, was connected to the gas line.

4.4. Digester Start-up and Feeding

Construction of the digester was completed in January 2010. Anaerobic sludge from a 1530 m^3 anaerobic digester (37.5 °C), fed with agricultural and food wastes, was used as inoculum. The first week of February, the digester was filled with a 1:1 ratio mixture of this inoculum and digestate from a small-scale anaerobic digester fed with dairy manure. Operation of the digester began in March 2010. The digester was fed three times per week with diluted dairy manure. The manure was first collected from the concrete surface of the dairy barns, and had an initial total solids content of 12%. The manure was then diluted with ground water to produce an average volatile solid (VS) content of 6% (SD = 0.89%), which matched the range of operation (6%–10%) suggested for this technology [42–44]. The amount of water added varied with the solid content of fresh manure; thus, the manure: water ratio ranged from 2:1 for dry manure to 10:1 for wet manure. The manure used had an average volatile solids (VS)/total solids (TS) ratio of 0.83 (SD = 0.04). This resulted in an average of 7.2% total solids. The organic loading rate ranged from 0.59 to 3.05 kg VS/m^3/day (Figure 2a). Based on the value of the organic loading rate, the following equation was used to calculate the volume of diluted manure to be fed:

$$Q = \left(\frac{OLR \times V}{C}\right) \times 1000 \times \frac{7}{3} \quad (1)$$

where:

Q = m^3 of diluted manure per fed (This volume ranged from 0.018 to 0.087 m^3 per load);

OLR = Organic Loading rate, kg VS/m^3/day;

V = 0.91 m^3 (240—digester working volume;

C = VS concentration, mg/L;

1000 × 7/3 = conversion factor to calculate the loading in m^3. The weekly organic loading rate was delivered three times per week.

After feeding the digester, a volume equivalent to the slurry fed was removed from the displacement tank, and ten gallons of slurry from the displacement tank were recirculated to the loading tank to increase mixing. Once a week, one gas sample was collected in a 0.5 L Tedlar bag, and liquid samples were taken in 250 mL plastic bottles from the loading tank, the digester chamber and the compensation tank. The gas sample was analyzed for CH_4 and CO_2. The liquid samples were analyzed for total solids (TS), VS, pH, and total volatile fatty acids (TVFAs). A one year of data collection was completed in March 2011 (363 days total).

4.5. Analytical Methods

TS and VS were determined gravimetrically following standard methods [45]. For total inorganic carbonate alkalinity (TIC), total volatile fatty acids (TVFAs) and total alkalinity, the raw sample was first centrifuged at 7500 rpm for 20 min. Then, the supernatant was diluted with DI water in a 1:3 ratio, and a total of 20 mL were titrated with H_2SO_4 0.1 N to end points of 5 and 4.4. The following empirical formulas were used for calculation [25]:

$$TIC = V_1 \times 250 \tag{2}$$

$$TVAS = \left((V_t - V_1) \times 1.66 - 0.15\right) \times 500 \tag{3}$$

where:

TIC = Total inorganic carbonate alkalinity, mg $CaCO_3$/L;
$TVAS$ = Total volatile fatty acids, mg HAc/L;
V_t = Total volume of H_2SO_4 0.1 N used, mL;
V_1 = Volume added from start to pH 5, mL;
$V_t - V_1$ = Volume added from pH 5 to pH 4.4, mL.

The composition of the biogas (CH_4 and CO_2) produced in the digester was quantified using a Shimadzu GC-14A gas chromatograph (Shimadzu Corporation, Kyoto, Japan) equipped with a thermal conductivity detector (TCD) and helium as the carrier gas. The calibration curve was developed with four standard gas (100% CO_2; 30% CO_2 and 70% CH_4; 70% CO_2 and 30% CH_4; and 100% CH_4). The R^2 of the calibration curve was higher than 0.999. A standard test was run with every ten samples. If the value returned for the standard deviated by more than three percent, a new calibration curve was developed.

4.6. Methane Yield

The methane yield was calculated according the Equation (4) using the average parameters of each stage showed in Table 1, as follows:

$$Methane\ Yield = \frac{\hat{B}\widehat{M}V}{\widehat{OLR}} \tag{4}$$

where:

$\hat{B}$ =avg. biogas production, L;
$\widehat{M}$ =avg. methane concentration, %;
V = Digester liquid volume, m^3;
$\widehat{OLR}$ = Avg. organic loading rate, kg VS/m^3/day.

4.7. Statistical Analysis

The operational data were grouped into five stages each corresponding to a different OLR (Table 2). The data for each stage (Table 1) represents a 95% confidence interval on the mean, which indicates that there is a 95% chance that this interval contains the true mean [24]. In addition, statistical differences of operational data among stages were assessed using unbalanced one-way Anova and the Tukey–Kramer multicomparison test [24]. A two-sample t-test was conducted to determine the mean VS concentration difference between the influent and effluent as a result of the digestion process [46]. These statistical analyses described were conducted using Matlab R2010.

Table 2. Digester operational stages. Operational data were split into five stages according the organic loading rate used and digester stability.

Stage	**Period**	**Justification**	**OLR, kg VS/m^3/day Mean (SD)**
(1) Start up	8 March 2010– 30 April 2010	Period after inoculation when the digester was first loaded	0.83 (0.12)
(2) Initial Rest	1 May 2010– 30 June 2010	Due to low gas production, the loading was stopped	0
(3) Optimal operation	1 July 2010– 10 November 2010	Operation with highest OLR and maximum biogas and methane production	2.43 (0.5)
(4) Poor Operation	11 November 2010– 10 January 2011	Operation with a lower OLR, the system showed signs of unstable performance in terms of decreasing total inorganic alkalinity and an increasing total volatile fatty acids content	1.58 (0.6)
(5) Final Rest	11 January 2011– 11 March 2011	Digester soured, loading was stopped	0

5. Conclusions

This study was an initial attempt to adapt a fixed-dome digester to meet the demand for small-scale digesters in small and medium size dairy farms in the U.S. Limitations and improvements that will be important while pursuing this technology for U.S. farms include the low methane yield obtained when temperatures drop below 20 °C, the need to better insulate the system, including the loading tank and the compensation tank. Additionally, the need for water to dilute the manure limit applications to water abundant areas. In the case of temperature, the digester can operate in climates similar to the Midwest U.S. for about six months (169 days), with digester temperatures above 20 °C, with acceptable methane yields (0.168 m^3/kg VS added) and no signs of inhibition. If digester temperatures fall below 20 °C, the organic loading rate should be lowered year round to keep the digester stable, but this management option will lower gas production throughout the year. Despite the limitations observed at temperatures below 20 °C, the results of this study demonstrate that burial of digesters, located in temperate regions, can moderate and increase digester temperature during the colder months. A complementary approach is to keep the digester temperature above 20 °C by heating the digester.

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Author Contributions

All authors contributed to the work in this manuscript. Martín was the principal investigator; he designed the pilot scale digester. Castano and Ciotola accomplished the sampling and lab work.

Castano conducted all the analysis and wrote the many drafts of this manuscript. Martin reviewed and revised the manuscript style.

Conflicts of Interest

The authors declare no conflict of interest.

References

1. Karim, K.; Klasson, K.T.; Drescher, S.R.; Ridenour, W.; Borole, A.P.; Al-Dahhan, M.H. Mesophilic digestion kinetics of manure slurry. *Appl. Biochem. Biotechnol.* **2007**, *142*, 231–242.
2. Weiland, P. Biogas production: Current state and perspectives. *Appl. Microbiol. Biotechnol.* **2010**, *85*, 849–860.
3. U.S. Environmental Protection Agency (USEPA). Anaerobic Digestion Capital Cost for Dairy Farms, 2010. Available online: http://www.epa.gov/agstar/documents/digester_cost_fs.pdf (accessed on 6 January 2011).
4. Demirer, G.; Chen, S. Two-phase anaerobic digestion of unscreened dairy manure. *Process Biochem.* **2005**, *40*, 3542–3549.
5. Parawira, W.; Read, J.S.; Mattiasson, B.; Björnsson, L. Energy production from agricultural residues: High methane yields in pilot-scale two-stage anaerobic digestion. *Biomass Bioenergy* **2008**, *32*, 44–50.
6. U.S. Environmental Protection Agency (USEPA). Operating Anaerobic Digester Projects, 2011. Available online: http://www.epa.gov/agstar/projects/index.html (accessed on 6 January 2011).
7. U.S. Department of Agriculture (USDA). Overview of the United States Dairy Industry, 2010. Available online: http://usda.mannlib.cornell.edu/usda/current/USDairyIndus/USDairyIndus-09-22-2010.pdf (accessed on 6 November 2011).
8. Daxiong, Q.; Shuhua, G.; Baofen, L.; Gehua, W. Diffusion and innovation in the Chinese biogas program. *World Dev.* **1990**, *18*, 555–563.
9. Khoiyangbam, R.S.; Kumar, S.; Jain, M.C.; Gupta, N.; Kumar, A.; Kumar, V. Methane emission from fixed dome biogas plants in hilly and plain regions of northern India. *Bioresour. Technol.* **2004**, *95*, 35–39.
10. Ciotola, R.J.; Martin, J.F.; Tamkin, A.; Castańo, J.M.; Rosenblum, J.; Bisesi, M.S.; Lee, J. The Influence of Loading Rate and Variable Temperatures on Microbial Communities in Anaerobic Digesters. *Energies* **2014**, *7*, 785–803.
11. He, P.J. Anaerobic digestion: An intriguing long history in China. *Waste Manag.* **2010**, *30*, 549–550.
12. Nianguo, L. Biogas in China. *Trends Biotechnol.* **1984**, *2*, 77–79.
13. An, B.X.; Preston, T. Gas production from pig manure fed at different loading rates to polyethylene tubular biodigesters. *Livest. Res. Rural Dev.* **1999**, *11*. Available online: http://www.lrrd.org/lrrd11/1/an111.htm (accessed on 20 August 2014).
14. Lettinga, G.; Rebac, S.; Zeeman, G. Challenge of psychrophilic anaerobic wastewater treatment. *Trends Biotechnol*. **2001**, *19*, 363–370.

15. McKeown, R.M.; Scully, C.; Enright, A.-M.; Chinalia, F.A.; Lee, C.; Mahony, T.; O'Flaherty, V.; Collins, G. Psychrophilic methanogenic community development during long-term cultivation of anaerobic granular biofilms. *ISME J.* **2009**, *3*, 1231–1242.
16. Lettinga, G.; Rebac, S.; Parshina, S.; Nozhevnikova, A.; van Lier, J.B.; Stams, A.J. High-rate anaerobic treatment of wastewater at low temperatures. *Appl. Environ. Microbiol.* **1999**, *65*, 1696–1702.
17. Rebac, S. Kinetics of fatty acid degradation by psychrophilically grown anaerobic granular sludge. *Bioresour. Technol.* **1999**, *69*, 241–248.
18. Rebac, S.; Lier, J.B.; Lens, P.; Cappellen, J.; Vermeulen, M.; Stams, A.J.M.; Lettinga, G.; Dekkers, F.; Swinkels, K.T.M. Psychrophilic (6–15 °C) high-rate anaerobic treatment of malting wastewater in a two-module expanded granular sludge bed system. *Biotechnol. Prog.* **1998**, *14*, 856–864.
19. Rebac, S.; Ruskova, J.; Gerbens, S.; Lier, J.B.; Stams, A.J.M.; Lettinga, G. High-Rate Anaerobic Treatment of Wastewater under Psychrophilic Conditions. *J. Ferment. Bioeng.* **1995**, *80*, 499–506.
20. Balasubramaniyam, U.; Zisengwe, L.S.; Meriggi, N.; Buysman, E. *Biogas Production in Climates with Long Cold Winters*; 2008. Wageningen University: Wageningen, The Netherlands. Available online: http://www.wecf.eu/english/publications/2008/biogas-coldclimates.php (accessed on 20 April 2010).
21. Hill, D.T.; Taylor, S.E.; Grift, T.E. Simulation of low temperature anaerobic digestion of dairy and swine manure. *Bioresour. Technol.* **2001**, *78*, 127–131.
22. Alvarez, R.; Lidén, G. Low temperature anaerobic digestion of mixtures of llama, cow and sheep manure for improved methane production. *Biomass Bioenergy* **2009**, *33*, 527–533.
23. Colleran, E.; Concannon, F.; Golden, T.; Geoghegan, F.; Crumlish, B.; Killilea, E.; Henry, M.; Coates, J. Use of methanogenic activity tests to characterize anaerobic sludges, screen for anaerobic biodegradability and determine toxicity thresholds against individual anaerobic trophic groups and species. *Water Sci. Technol.* **1992**, *25*, 31–40.
24. Ho, R. *Handbook of Univariate and Multivariate Data Analysis and Interpretation with SPSS*; CRC Press: Boca Raton, FL, USA, 2006.
25. Lossie, U.; Pütz, P. Targeted control of biogas plants with the help of FOS/TAC. In *Practice Report, Laboratory Analysis, Titration FOS/TAC*; Hach Lange Ltd.: Salford, UK, 2001.
26. Ward, A.J.; Hobbs, P.J.; Holliman, P.J.; Jones, D.L. Optimisation of the anaerobic digestion of agricultural resources. *Bioresour. Technol.* **2008**, *99*, 7928–7940.
27. Kanwar, S.S.; Gupta, R.K.; Guleri, R.L.; Singh, S.P. Performance evaluation of a 1 m^3 modified, fixed-dome Deenbandhu biogas plant under hilly conditions. *Bioresour. Technol.* **1994**, *50*, 239–241.
28. Kalia, A.; Kanwar, S. Long-term evaluation of a fixed dome Janata biogas plant in hilly conditions. *Bioresour. Technol.* **1998**, *65*, 61–63.
29. Martin, J., Jr.; Wright, P.; Inglis, S.; Roos, K. Evaluation of the performance of a 550 cow plug-flow anaerobic digester under steady-state conditions. In *Animal, Agricultural, and Food Processing Wastes IX*; Joseph, St., Ed.; American Society of Agricultural Engineers: Triangle Park, NC, USA, 2003.
30. Callaghan, F.; Wase, D.; Thayanithy, K.; Forster, C. Continuous co-digestion of cattle slurry with fruit and vegetable wastes and chicken manure. *Biomass Bioenergy* **2002**, *22*, 71–77.

31. Schoen, M.A.; Sperl, D.; Gadermaier, M.; Goberna, M.; Franke-Whittle, I.; Insam, H.; Ablinger, J.; Wett, B. Population dynamics at digester overload conditions, *Bioresour. Technol.* **2009**, *100*, 5648–5655.
32. Safley, L., Jr.; Westerman, P. Low-temperature digestion of dairy and swine manure. *Bioresour. Technol.* **1994**, *47*, 165–171.
33. Van Horn, H.H.; Wilkie, A.C.; Powers, W.J.; Nordstedt, R.A. Components of Dairy Manure Management Systems. *J. Dairy Sci.* **1994**, *77*, 2008–2030.
34. Kaparaju, P.L.N.; Rintala, J. The effects of post-treatments and temperature on recovering the methane potential of >2 mm solid fraction of digested cow manure. *Environ. Technol.* **2005**, *26*, 625–632.
35. Guo, J.; Li, X.; Xu, P.; Dong, R.; Clemens, J. Process control and fluxes of medium size agricultural biogas plants management at ambient temperature: A case study in Beijing. Available online: http://www.ramiran.net/ramiran2010/docs/Ramiran2010_0279_final.pdf (accessed on 6 July 2011).
36. Hashimoto, A.; Varel, V.; Chen, Y. Ultimate methane yield from beef cattle manure: Effect of temperature, ration constituents, antibiotics and manure age. *Agric. Wastes* **1981**, *3*, 241–256.
37. Kalia, A. Development and evaluation of a fixed dome plug flow anaerobic digester. *Biomass* **1988**, *16*, 225–235.
38. Lehtomäki, A.; Huttunen, S.; Rintala, J.A. Laboratory investigations on co-digestion of energy crops and crop residues with cow manure for methane production: Effect of crop to manure ratio. *Resour. Conserv. Recycl.* **2007**, *51*, 591–609.
39. Food and Agriculture Organization (FAO). Water Lifting. FAO Irrigation and Drainage. Paper 43; 1986. Available online: http://www.fao.org/docrep/010/ah810e/AH810E13.htm (accessed on 20 January 2012).
40. Ciotola, R.J.; Martin, J.F.; Castańo, J.M.; Lee, J.; Michel, F. Microbial Community Response to Seasonal Temperature Variation in a Small-Scale Anaerobic Digester. *Energies* **2013**, *6*, 5182–5199.
41. Desai, S.R.; Palled, V. Performance evaluation of fixed dome type biogas plant using solid state digestion of cattle dung. *Environ. Ecol.* **2013**, *31*, 435–439.
42. Rajendran, K.; Aslanzadeh, S.; Taherzadeh, M.J. Household biogas digesters—A review. *Energies* **2012**, *5*, 2911–2942.
43. Lorimer, J.; Powers, W.; Sutton, A. *Manure Characteristics*; Midwest Plan Service: Ames, IA, USA, 2004.
44. American Public Health Association. *2005 Standard Methods for the Examination of Water and Wastewater*, 21 Har/Cdr ed.; American Public Health Association: Washington, DC, USA, 2005.
45. Ramsey, F.L.; Schafer, D.W. *The Statistical Sleuth: A Course in Methods of Data Analysis*; Duxbury Press: Belmont, CA, USA, 1997; pp. 100–150.
46. Zeeman, G.; Sutter, K.; Vens, T.; Koster, M.; Wellinger, A. Psychrophilic digestion of dairy cattle and pig manure: Start-up procedures of batch, fed-batch and CSTR-type digesters. *Biol. Wastes* **1988**, *26*, 15–31.