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# Performance Assessment of Solid State Anaerobic Digestion of Poultry Litter

# **Mason Christopher Puckett**

Biological Engineering Program

Biological and Agricultural Engineering Department

College of Engineering

University of Arkansas

Undergraduate Honors Thesis

### **ADVISORY AND COMMITTEE SIGNATURE PAGE**

This thesis has been approved by the Biological and Agricultural Engineering Department for submittal to the College of Engineering and Honors College at the University of Arkansas.

Muny	_Signature	4/26/2018 Date		
Wen Zhang, BENG Honors Advisor				
Bey S	_Signature	04/26/2018 Date		
Brian E. Haggard, Honors Committee Member				
Sylve	_Signature	4/26/18 Date		
Jun Zhu, Honors Committee Member				
Thomas of he	_Signature	4/26/18 Date		
Thomas Costello, Honors Committee Member		1		
BS AS	_Signature	04/26/2018 Date		
Departmental Honors Coordinator or Department Head				

#### Abstract

The disposal of poultry litter can exert an economic and environmental burden to the agriculture community. As a result, it is desirable to reduce the amount of waste and recover resources from the waste. This study focuses on the construction and preliminary testing of a laboratory scale (20 L) solid state anaerobic digester (AD) fed with dry poultry litter. Glucose was added in addition to the poultry litter to achieve the appropriate C:N ratio to support the growth of anaerobic microorganisms. The AD was first fed every 4 days at 4 g VS/L/feeding for 24 days, rested (no feeding) for 32 days, and then at 8 g VS/L/feeding every 4 days for 24 days, followed with 33 days of no feeding. During the experiment the following parameters were measured: total solids (TS), volatile solids (VS), total nitrogen (TN), total carbon (TC), chemical oxygen demand (COD), pH, and biogas yield and composition. Throughout the experiment, as the litter accumulated in the AD, TS, VS, TC, TN, and COD all increased gradually. pH however showed a dramatic decrease to 5.2, which is likely the main reason for the low biogas yield and near zero CH<sub>4</sub> production. It is recommended to closely monitor pH and buffer it to a near neutral range to sustain the growth of methanogens.

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#### Introduction

Arkansas, due to its large poultry industry, produces 1.3 million metric tons of poultry litter annually [1]. Poultry litter refers to a mixture of manure and an organic material used for absorbing moisture and containing odors. Sawdust, paper, and rice hulls are commonly used as the organic material [2]. Litter is traditionally land-applied as fertilizer due to its high plant nutrient (N and P) content [3]. However, application of litter in proportion to the N requirement of crops has led to the buildup of P in soils and eventually large quantities of P in runoff. This leads to eutrophication of water bodies and other negative environmental impacts [4]. Due to poultry litter's low N:P ratio and the high concentration of poultry production in northwest Arkansas, legislation was proposed in 2003 requiring poultry producers to export their excess litter outside the region [5]. Litter export is an economic burden on producers and does not address the problem of soil P and water pollution long-term. Therefore, other outlets/uses for the litter must be found to sustainably meet the needs of the agricultural community.

Several methods exist to locally address litter disposal and usage. One method is direct combustion of the litter for heating and power generation [6]. While this was a very attractive idea originally, research later showed that emissions from incineration of litter included particulate matter, bio-aerosols, arsenic, and various other toxins, which could be hazardous to human health [4, 7]. Additionally, most of the poultry production is located around low-income communities that are already vulnerable to disease due to poorer access to medical care [8].

Composting the litter is an attractive alternative as a somewhat quick way of disposal of litter [9, 2]. Composting litter, however, decreases the N content, further decreasing the N:P ratio, and therefore decreases the litter's economic value as a fertilizer. Additionally, composting large amounts of litter is expensive, requiring lots of equipment, labor, and acreage [10].

Lastly, anaerobic digestion of poultry litter is an alternative. Anaerobic digestion is the process by which an organic material is put under heated anaerobic conditions to encourage the growth of anaerobic

methanogenic bacteria [11]. The bacteria under these conditions produce methane, a clean-burning renewable fuel. Additionally, this process ideally keeps the litter from losing its value as a fertilizer maintaining its nutrient content [2], while significantly decreasing the pathogen (fecal coliforms, fungal spores, salmonellae, etc.) levels within the effluent due to the anaerobic conditions [12]. However, poultry litter poses a challenge to the digestion process due to its low moisture content, and often requires copious amounts of water to convey and maintain anaerobic conditions. Solid state digestion is a possible solution to this problem, even while still requiring a moisture content of 75% [13]. Solid state digestion consists of an anaerobic digester (AD) with a bed of solid material at the bottom in which methanogenic bacteria can grow.

One of the main goals of this project was to construct an AD and establish an efficient method for delivering the waste into the AD without using large quantities of water, as is the industrial practice. Additionally, this study seeks to provide preliminary data on the effect of litter loading rate into the AD, by varying the litter loading rate and observing the effect on gas production in composition and volume.

#### Methods

#### **Construction:**

The AD was constructed from a 112-cm long section of clear 15.24-cm i.d. PVC pipe by capping the ends and sealing (see Figure 1). Several sampling ports for both gas and liquid samples were installed along the body of the AD. The AD was wrapped in an automated water jacket to sustain the temperature at 32 °C. A Caframo SSM31 stir rod was used to ensure constant mixing. On the top cap, a gas port was placed and attached to a 3 L Tedlar bag and an Archae Press wet tip gas meter. To insure the AD was sufficiently sealed, the digester was pressurized to 2 psi overnight. If the pressure dropped significantly in that time, the leaks were plugged, and the pressure test repeated until no significant change in pressure was observed.

A variety of feeding mechanisms was tested. The first method of feeding consisted of a PVC tube with a drill powered plunger pushing the dry litter through the tube into the AD through a ball valve. This method failed due to the high amounts of friction that the litter created when compressed in the tube. To decrease the friction, the ball valve connecting the feed tube with the AD was replaced with a gate valve. However, this too produced too much friction. Due to the failures of the plunger system, a liquid driven feeding mechanism was constructed which successfully conveyed the litter.

The liquid driven feeding mechanism works by circulating the liquid in the AD through the litter in an air-evacuated chamber (feeding tube in Figure 1) and back into the AD, pulling the litter with it. The mechanism consists of a clear PVC vertical tube attached to the bottom of the AD via a rubber elbow connector and a gate valve. During feeding, a peristaltic pump circulates water from a sampling port midway up the AD, into the litter-loaded chamber, through the elbow and gate valve, and back into the bottom of the AD. This allows the litter to accumulate at the bottom, providing a solid "bed" in which methanogenic bacteria can grow.

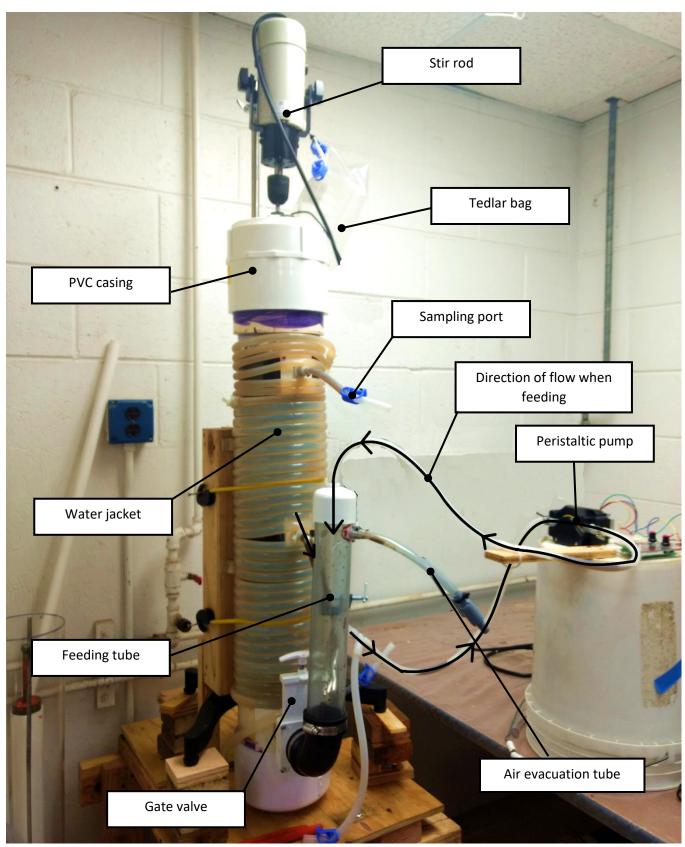


Figure 1: Anaerobic digester

#### Startup and Feeding:

The AD was started with 530 g of dry litter (composition shown below in Table 1) and filled to 15 L resulting in an initial sludge composition of 2.5% TS. The initial TN, TC, and TS of the dry litter were measured by the Agriculture Diagnostics Lab using methods shown in Table 1 to determine the initial concentrations of the N and C as well as the composition of total solids. The results of these measurements are shown below in Table 1. Additionally, the VS components were measured using the standard method 2540 E [14] finding the dry litter's VS composition to be 70.10% of the TS.

Table 1: Composition of source poultry litter

Parameter	Prevalence (% dry litter)	Measurement Method
H₂O	26.70	Standard Methods 2540 B [15]
solids	73.30	Standard Methods 2540 B [15]
С	27.08	Elementar vario MAX CN analyzer
N	2.79	Elementar vario MAX CN analyzer
Ca	2.49	Atomic absorption spectroscopy [16]
K	2.61	Atomic absorption spectroscopy [16]
Р	1.60	Atomic absorption spectroscopy [16]
NH <sub>4</sub> -N	0.4028	Colorimetry with Spectro auto Analyzer [17]

Over the 111 days of the experiment, the digester was fed at rate of 1 g VS/L/day (4 g VS/L/feeding, fed on average every 4 days) on days 0-24, followed by days 25-58 of no feeding. Then the second feeding rate of 2 g VS/L/day (8 g VS/L/feeding, fed on average every 4 days) was used on days 59-83 followed by days 84-111 of no feeding. During times of feeding the digester gas (when produced) and sludge was sampled immediately following every feeding. During times of no feeding the gas and sludge were sampled every 4 days on average.

With a desired VS loading rate and known TS and VS compositions, the litter loading rate (LLR) was calculated in the following equation:

$$LLR = VS \ g/L/day \times \frac{1}{\% \ TS} \times \frac{1}{\% \ VS} \times [volume] \ L \times [time \ since \ last \ feed] \ day$$
 
$$LLR_1 = 1.0 \ g/L/day \times \frac{1}{73.30\%} \times \frac{1}{70.10\%} \times 14.45 \ L \times 4 \ day = 112.49 \ g \ litter/feeding$$
 
$$LLR_2 = 2.0 \ g/L/day \times \frac{1}{73.30\%} \times \frac{1}{70.10\%} \times 14.45 \ L \times 4 \ day = 224.98 \ g \ litter/feeding$$

The litter's default C:N ratio from the measurements in Table 1 can be determined through the following calculation:

$$\frac{C}{N} = \frac{Total \% C}{Total \% N} = \frac{27.08\%}{2.79\%} = 9.70 C: N$$

By recommendation from Dr. Zhu, the desirable C:N ratio is between 15:1 and 30:1. Therefore, 50% of the litter added by mass was matched by mass of glucose. This resulted in a C:N ratio of 16.95, falling within the appropriate range.

#### Measurements:

The measured parameters within the sludge are as follows: TS, VS, TN, TC, and COD. The TS and VS of the sludge samples were measured using standard methods 2540 B [15] and 2540 E [14], respectively, using a Binder laboratory oven and a Cole-Parmer StableTemp ® muffle furnace. The TN was measured using Hach TNT 827 TN kit using a x40, x80, or x100 dilution of the samples as needed to keep within the test's range [18]. The COD was measured using a Hach TNT 823 COD kit using a x10 and x20 dilution of the samples to keep the tests within range [19]. TC was measured using an Elementar varioMAX CN analyzer by the University of Arkansas Agricultural Diagnostic Laboratory.

Samples of biogas were analyzed with a GC-2014 (Shimadzu, Japan) gas chromatographer. The instrument used helium as the carrier gas at 40 mL/min of flow, a packed metal column (ST 2m length, 2mm i.d.) and thermal conductivity detector. Temperature started at 40 °C, ramping to 95 °C at 25 °C per minute. Volumetric analysis of the gas produced was conducted using a CX-XML/XMF wet gas portable air flow meter.

Only one sludge sample and one gas sample were taken from the AD on each sampling day.

Linear regression was conducted on each subset of data to estimate the rate of change of each parameter during feeding times (days 0-24 and 59-83) and times of no feeding (days25-58 and 84-111).

The trendlines with their respective slopes and R<sup>2</sup> values are shown on Figures 2-7.

#### **Results and Discussion**

#### Sludge Analysis:

The following data was collected over the digestion time with the first feeding rate of 1 g VS/L/day on days 0-24 followed by days 25-58 of no feeding and the second feeding rate of 2 g VS/L/day on days 59-83 followed by days 84-111 of no feeding.

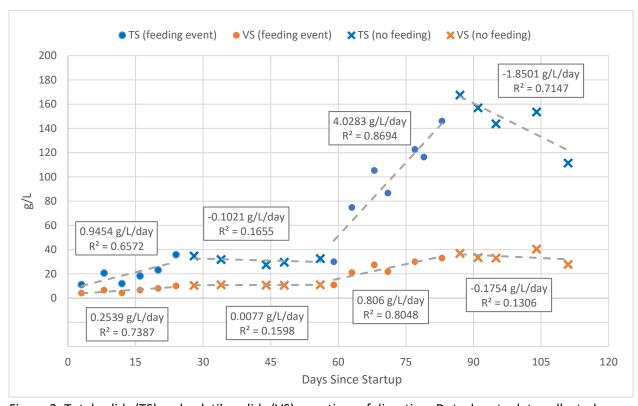


Figure 2: Total solids (TS) and volatile solids (VS) over time of digestion. Dots denote data collected on days when the digester was fed, while x's denote samples taken during times of no feeding. Rates in boxes denote the slopes of the adjacent trendline.

As shown in Figure 2, TS concentrations accumulated in the digester over time at estimated rates of 0.9454 g/L/day (first feeding period) and 4.028 g/L/day (second feeding period). VS concentrations accumulated at estimated rates of 0.2539 g/L/day (first feeding period) and 0.806 (g/L/day). As expected with an increase in loading rate, the rate of solids accumulation also increased after the feeding rate was changed to 2 g/L/day as shown by the increase in estimated slopes by a factor of 4 from first feeding period to second feeding period. According to several reports, summarized in K. Singh et al, the upper limit of productive anaerobic digestion is approximately 10% TS (approx. 100 g/L). One study using poultry

manure reported a TS concentration of 21.7% (217 g/L), which completely inhibited anaerobic digestion [20]. Additionally according to one study [21], digestion under higher TS concentrations tended toward lower pH, which is detrimental to bacterial activity.

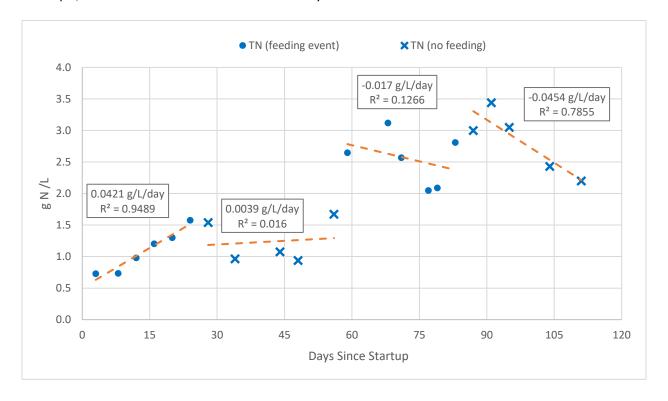


Figure 3: Total nitrogen (TN) over the time of digestion. Dots denote data collected on days when the digester was fed, while x's denote samples taken during times of no feeding. Rates in boxes denote slopes of the adjacent trendline.

As shown in Figure 3, the TN of the sludge across the first VS loading rates initially and increased with the accumulation of the litter. The TN increased during the first period of feeding (slope of 0.041 g N/L/day), plateaued during the first period of no feeding (slope decreased by a factor of 10), then did not increase as expected during the second period of feeding (slope of -0.017 g/L/day), and then decreased (estimated slope of -0.0454 g/day) during the second period of no feeding. This drop means that N was leaving the system, most likely to some gaseous form in the biogas produced. Further testing is required to determine the N species leaving the system.

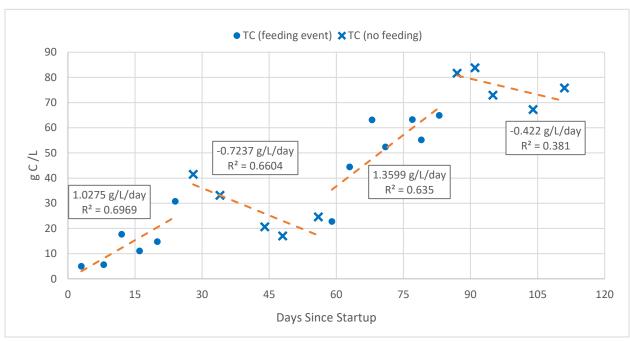


Figure 4: Total carbon (TC) over the time of digestion. Dots denote data collected on days when the digester was fed, while x's denote samples taken during times of no feeding. Rates in boxes denote slopes of the adjacent trendline.

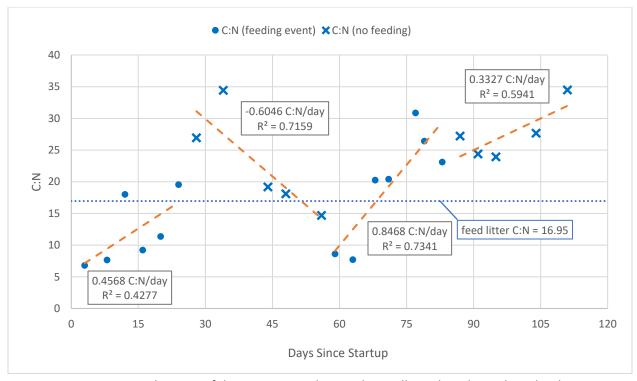


Figure 5: C:N ratio over the time of digestion. Dots denote data collected on days when the digester was fed, while x's denote samples taken during times of no feeding. Rates in boxes denote slopes of the adjacent trendline.

In anaerobic digestion, a proper C:N ratio is extremely important to the production of biogas, as excess nitrogen can lead to toxicity from free ammonia which inhibits bacterial growth. Generally a C:N ratio from 15:1 to 30:1 is optimal, which is why the digester was fed with a mixture of glucose and dry litter. However, the startup mixture did not include any glucose to supplement the carbon, which accounts for the low starting C:N ratio.

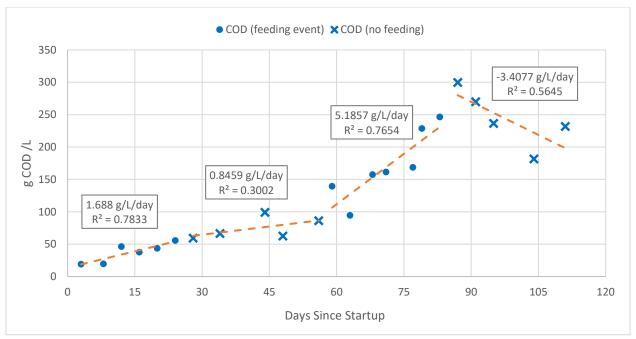


Figure 6: Chemical oxygen demand (COD) over the time of digestion. Dots denote data collected on days when the digester was fed, while x's denote samples taken during times of no feeding. Rates in boxes denote slopes of the adjacent trendline.

As shown in Figure 6, the COD increased from days 3-83 with positive regression slopes of 1.688, 0.846, and 5.187 g/L/day. The rate of increase of COD during a VS loading rate 2 g VS/L/day was 3 times that of the COD rate of increase during the VS loading rate of 1 g VS/L/day. This may be due to the unfavorable acidic conditions during days 59-83 which decreased bacterial activity and decreased COD reduction. However, during the periods of no feeding (days 28-56 and 87-111), the COD seemed to be more sporadic and sharply declined during the second period of no feeding (days 87-111). The steady increase in COD over the course of the digestion time, indicates a lack of bacterial activity as the COD reduction is a common measure of bacterial activity [20].

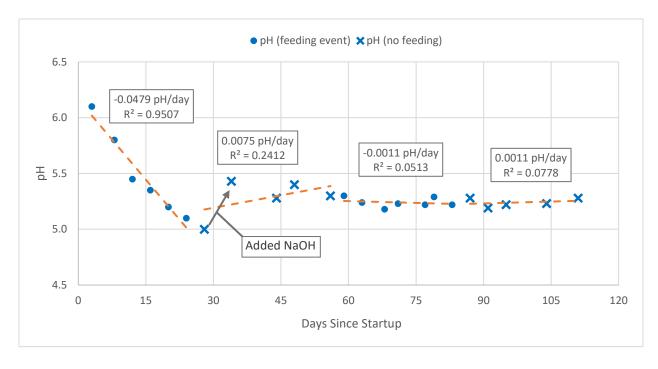


Figure 7: pH over the time of experiment. Dots denote data collected on days when the digester was fed, while x's denote samples taken during times of no feeding. Rates in boxes denote slopes of the adjacent trendline.

Figure 7 displays the change in pH over the experimental period. During the first 28 days of operation, the pH decreased from 6.1 to 5.0. This was lower than the desired pH range for anaerobic digestion. For methanogenesis, the pH must be kept relatively close to neutral, with the optimal pH being in the range of 6.8 to 8.5 [20]. An attempt was made to boost the pH after measuring the low value of 5.0 on day 28 by adding NaOH solution. The pH increased to 5.43 on day 34 and remained above 5.18 throughout the rest of the experiment. The decrease in pH can be an indication of metabolic activity by acidogenic bacteria. While necessary in anaerobic digestion, the excess decrease in pH can create unfavorable conditions for the methanogenic bacteria. It is believed the low pH was the main reason that methane production was near zero in this experiment (see Figure 8).

#### Gas Analysis:

The gas analysis showed that the biogas produced was mainly comprised of CO<sub>2</sub>, CH<sub>4</sub>, and N species. The concentration of CO<sub>2</sub> and CH<sub>4</sub> in the biogas produced over time is shown in Figure 8. As the CO<sub>2</sub> production fluctuates, no methane production was detected except in the sample from day 79. This measurement of 0.8% CH<sub>4</sub> on day 79 is close to the lower limits of the gas chromatographer's range, which explains why there is little to no data for the composition of CH<sub>4</sub>. This is indicative of underdeveloped methanogen cultures in the AD. The suppression of methanogens is most likely the result of an overly acidic environment. The composition of higher CO<sub>2</sub> in the gas occurred during times of COD reduction as shown in Figure 6, especially during the second period of no feeding.

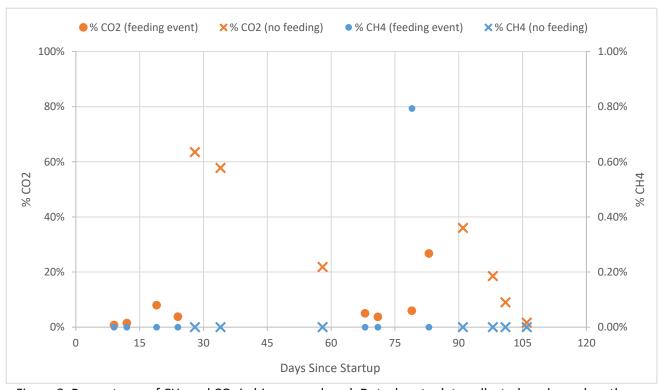


Figure 8: Percentages of  $CH_4$  and  $CO_2$  in biogas produced. Dots denote data collected on days when the digester was fed, while x's denote samples taken during times of no feeding.

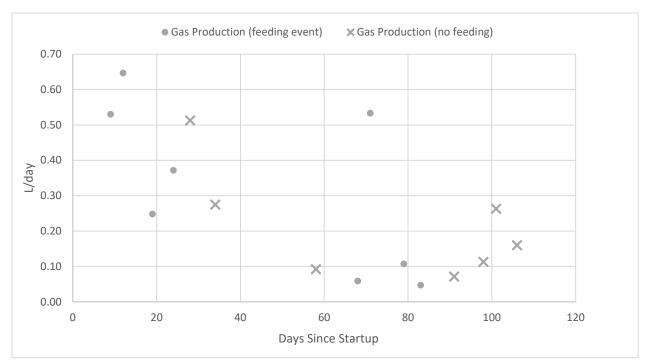


Figure 9: Biogas production. Dots denote data collected during periods when the digester was fed, while x's denote samples taken during times of no feeding.

Using the following equation below developed by Webb and Hawkes [22] for the expected volume of biogas produced per gram of VS, the expected gas production rate was determined based on the retention time and influent substrate concentration:

gas yield 
$$L/g$$
  $VS = K_C \left(1 - \frac{K_S(D + K_d)}{S_0(\mu_{max} - D - K_d)}\right)$ 

Where  $K_C$  is the rate constant of 0.400 L/g.  $K_S$  and  $\mu_{max}$  are constants in the Monrod equation and are 8.933 L<sup>-1</sup> and 0.326 day<sup>-1</sup> respectively.  $K_d = 0.1 * \mu_{max}$ .  $S_0$  is the influent substrate concentration (g VS/L), and D = 1/retention time (day<sup>-1</sup>). [20]

For loading rates of 1 and 2 g VS/L/day the expected gas yield was 3.26 and 9.11 L/day respectively. This expected value is much greater than the observed gas yield, shown in Figure 9, and the expected trend of a higher gas yield with a higher VS loading rate was not observed. This could be, in part, because the excess TS and low pH inhibited the anaerobic bacterial activity.

For future iterations of the experiment, regular pH monitoring and the addition of buffers such as bicarbonate to keep the pH within favorable range (between 6.5 and 8.5) is recommended. This should

encourage a favorable proportion of acidogenic and methanogenic bacteria to facilitate methanogenesis.

Additionally, the quality of litter used for feeding should be checked routinely to ensure the VS and TS composition remains stable throughout the experiment, and the litter loading rate should be adjusted according to any significant deviation in the %VS.

#### Conclusion

The performance of a solid state AD using poultry litter was tested. To deliver the litter, several feeding mechanisms were designed and tested. This resulted in the use of a liquid driven feeding mechanism that recirculates the AD's fluid to flush the litter into the AD. To test the AD's performance, poultry litter was added every 4 days for 24 days and let rest for 30 days afterwards. Two VS loading rates were tested sequentially. During the startup of the digester, the pH decreased rapidly over the first few days, and over the course of the experiment, the pH dropped from 6.5 and leveled out to about 5.2, which is well below the preferred range of methanogenic bacteria. Throughout the experiment the CH<sub>4</sub> yield was negligible in all samples except for one (day 79). The accumulation of COD, low pH decrease, and lack of CH<sub>4</sub> yield indicates there was an overgrowth of acidogenic bacteria and no sustained culture of methanogens. The effect of VS loading rate on gas composition and yield was not able to be determined due to the confounding variable problem of low pH. For future experimentation the pH should be closely monitored to remain at neutral range for sustaining the methanogens in the digester.

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