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A scale-up procedure for substrate co-digestion in anaerobic digesters through the use of substrate characterization, BMPs, ATAs, and sub pilot-scale digesters

by

Steven Thomas Sell

A thesis submitted to the graduate faculty

in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

Co-majors: Agricultural Engineering; Biorenewable Resources and Technology

Program of Study Committee:
Robert T. Burns, Co-major Professor
D. Raj Raman, Co-major Professor
Robert P. Anex

Iowa State University

Ames, Iowa

2011

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ACKNOWLEDGEMENTS

I would first like to thank Dr. Robert T. Burns for making this thesis possible by selecting me as a graduate research assistant. Through the research process I have been able to pursue my passion for solid waste engineering and have learned a great deal from him. I am very fortunate that Dr. D. Raj Raman was willing to step in and give me direction and guidance as my graduate career unfolded. With the help of Dr. D. Raj Raman and Dr. Robert P. Anex my graduate education was expanded beyond waste engineering, for that I am grateful.

The data and results found within this paper would not have been possible without the assistance of fellow research assistants, visiting scholars, and undergraduate students within the Agricultural Waste Management Laboratory at Iowa State University. For their contributions I would like to thank Lara Moody, Ross Muhlbauer, Daniel Andersen, Randy Swestka, Brad Bond, Laura Pepple, Olusoji Adeyosoye, Gayle Bishop, Jacob Baker, John Stinn, Brett Gutknecht, Jenae Baumert, Amy Sroka, Sarah Johnson, Daniel Puck, and J. Wyatt Hall.

ABSTRACT

The main objective of this thesis was to develop a system for predicting methane production and anaerobic digestion performance of multiple substrates prior to implementation of full-scale application. This thesis is prepared in the journal paper format and includes three papers that were prepared for submission to a journal or conference proceedings.

The objective of the first paper was to analyze multiple substrates using various laboratory techniques so that optimum mixture ratios could be formed. Biochemical methane potentials (BMPs) and anaerobic toxicity assays (ATAs) were used to select and in some cases rule out substrates based on their contribution to methane production. Mixtures were created using constraints arising from the full-scale system. This included the use of all available manure, keeping total solids below 15% to facilitate pumping, maintaining pH between 6.5 and 8.2 for microbial ecology, providing high COD concentrations to maximize methane production, and limiting ammonia levels to avoid toxicity (Speece, 1996). The BMP and ATA results from each mixture were analyzed and compared. The mixture with the best performance was selected for subsequent testing in 100-L sub pilot-scale anaerobic digesters.

The objective of the second paper was to analyze the performance of three 100-L sub pilot-scale anaerobic digesters. These plug flow digesters operated at a 21-d hydraulic retention time (HRT) and were fed the mixture selected in the first paper in a semi-continuous manner twice weekly (6 loadings per HRT). Methane production was measured using submerged tipping buckets. Methane production from the sub-pilot

scale reactors was compared to that predicted by the BMP tests. After two hydraulic retention times, the BMP maximum and minimum were observed to be valid boundaries for the sub-pilot scale anaerobic digester methane production, with some of the variability ascribed to seasonal substrate changes.

The objective of third and final paper was to use a series of BMPs and an ATA to predict the methane production in three 100-L sub pilot-scale anaerobic digesters that were subjected to a potential toxicant, glycerin. A group of ATAs were performed with glycerin inclusion rates of 0.5%, 1.0%, 2.0%, 4.0%, 8.0%, 15%, 25%, and 35% by volume. A set of BMPs was performed where a baseline mixture was combined with glycerin such that glycerin was 0.0%, 0.5%, 1.0%, 2.0%, 4.0%, 8.0%, 15%, 25%, and 35% of the combined mixture by volume. In addition, BMPs of 100% glycerin and 50% glycerin/50% DI water by volume were also performed. The three 100-L sub pilot-scale anaerobic digesters were operated at a 21-d hydraulic retention time (HRT) and were each fed in a semi-continuous manner twice weekly (6 loadings per HRT). Each digester was fed a combination of the mixture selected in paper one with a different amount of glycerin (1%, 2%, 4% by volume). The ATAs showed that glycerin was toxic to methane production at all inclusion levels. The BMPs indicated no significant difference between methane production of the 0.0%, 0.5%, 1.0%, 2.0%, and 4.0% mixture combinations; however, at 8.0%, methane production tripled. In contrast, the sub pilot-scale reactors showed signs of toxicity 4.0% glycerin inclusion and little to no effect on methane production for 1.0% and 2.0% glycerin inclusion. Thus, neither the ATA nor the BMP proved to be an adequate predictor for the sub pilot-scale reactors.

The most likely cause was lack of mixing within the sub pilot-scale digester to keep glycerin suspended and the mixture well blended. The separation of materials probably lead to short circuiting and prevented adequate microbial activity and methane formation.

CHAPTER 1. GENERAL INTRODUCTION

Background and Justification

The United States is home to more than 450,000 animal feeding operations (AFOs) (USDA NRCS, 2009). As the number of AFOs continues to increase, and stocking densities continue to rise, the regulations controlling each operation become more stringent. Regulations such as comprehensive nutrient management plans (CNMPs) have been established to monitor feed, manure and urine, dead animals, and farm safety (USDA NRCS, 2011).

Proper manure management strategies vary by species, number of animals, region, and economics. In all cases, the treatment of manure helps reduce eutrophication of receiving waters, odor emissions, and other air-pollutant emissions (Carucci et al. 2005). Current methods used to treat and/or dispose of manure include: land application, lagoon systems, ground injection, constructed wetlands, reverse osmosis, and anaerobic digestion (Gungor-Demirci & Demirer, 2004). Each strategy differs in terms of mitigation efficiency and environmental impact. Selection of a treatment process is based largely on the ability of a system to fit the socioeconomic needs of the operation and surrounding region.

Anaerobic digestion (AD) systems have the potential to alleviate some dependence on fossil fuels in the United States through the generation of electricity from the combustion of more than 50,000 metric tons methane annually (US EPA AgStar, 2010a). Reductions in greenhouse gas (GHG) emissions and pathogens are also benefits of AD (USDA NRCS, 2007). AD systems have been shown to be reliable

and economically successful in numerous cases (De Baere 2000; Ten Brummeler 2000; Mata-Alvarez et al. 2000). However, there has been limited long-term success of AD systems within the United States mostly attributed to poor system design, installation, and management. Yet, in systems that have ceased operation, the main cause was not technology but rather operation and maintenance costs (USDA NRCS, 2007). The United States Environmental Protection Agency (EPA) AgSTAR reports that there are currently 151 operational AD projects producing 392,000 MWh/yr equivalents in the U.S. There have been numerous financial incentives from the USDA and energy independence organizations to construct and operate these facilities (US EPA AgSTAR, 2010b). Financial incentives help offset the high capital and operating costs of AD systems; however, investment into a full-scale operation can be quite costly.

A model created by Faulhaber et al. (2011) suggests that because of the relatively low commercial energy prices in the US, for a dairy cattle plug flow digester approximately 1,000 head or larger is needed to meet a positive payback. However, higher revenues are possible by increasing the organic loading rate of the digester, for example by using a mixed waste stream. By co-digesting manure with high organic waste streams such as industrial wastewaters, both industry and farms benefit. With the combination of multiple wastes comes the challenge of maintaining proper chemical and biological activity as well as physical handling issues. Performing co-digestion studies at full-scale can be quite risky and lead to failure. Therefore, small or pilot-scale work to prove performance and operation is useful prior to full-scale implementation to prevent costly full-scale failure.

Objectives

The objective of this thesis is to formalize a testing process that will start with the identification of possible anaerobic digestion co-substrates, use bench-scale tests to select from among substrates, and lead to sub pilot-scale digester application. Specifically this thesis compares the performance of Biochemical Methane Potential (BMP) Assays and Anaerobic Toxicity Assays (ATAs) with the results obtained from sub pilot-scale anaerobic digesters. The goals that this research aimed to achieve were as follow:

- To formulate a library of possible co-digestion substrates that were available to Amana Farms, Inc.
- To analyze substrate performance in BMPs and ATAs both individually and in mixtures
- To compare mixture BMPs and ATAs of mixture to sub pilot-scale reactors.

This thesis work was primarily funded by a grant targeted to assist the Amana Farms anaerobic digester in eastern Iowa. Funding was provided by The Iowa Office of Energy Independence and Amana Farms, Inc. The grant required that Iowa State University help Amana Farms optimize the full-scale digester performance, assist with improving operation and maintenance capabilities, and assist in the preparation of the educational extension programs requirement of Amana Farms, Inc. by the Iowa Office of Energy Independence.

Optimizing the full-scale digester's performance entailed stabilizing methane production and analyzing the digester's situation. Then BMPs and sub pilot-scale

digesters were used to analyze various substrate mixture combinations in order to select successful mixtures for potential use at full-scale implementation.

Improving operation and maintenance capabilities was necessary to allow Amana Farms, Inc. to independently monitor and diagnose full-scale digestion issues. Facility operators were shown standard operating procedures and parameters to monitor for successful operation.

Preparation of the educational extension programming requirements of Amana Farms Inc. was needed in order to fulfill the requirements of the Iowa Office of Energy Independence. These extension events were open to the public to display the knowledge gained during the operation of the Amana Farms' digester. Information was provided during three half-day conferences with technical presentations by subject experts.

Thesis Organization

This thesis contains a general introduction, three research articles, a general conclusion, and three appendices. The general introduction includes the justification and objectives of this thesis and a brief literature review.

The first article, entitled "Approaches for Selecting Anaerobic Digestion Co-Substrates for a Full-Scale Beef Manure Digester Using Biochemical Methane Potentials and Anaerobic Toxicity Assays," was published in the conference proceedings of the 2010 American Society of Agricultural and Biological Engineers (ASABE) International Symposium on Air Quality and Manure Management for Agriculture held in Dallas, Texas. This article gave a summary of how BMPs and ATAs

were used to narrow down substrates for anaerobic digestion and create mixtures for co-digestion.

The second article, entitled “Comparison of Methane Production from Bench- and Sub Pilot-Scale Anaerobic Digesters,” was submitted to *Applied Engineering in Agriculture.* This article compared the performance of a co-digestion mixture in bench-scale BMPs to sub pilot-scale anaerobic reactors.

The third research article entitled “Anaerobic Co-Digestion of Mixed Substrates: Relations between Bench-Scale Assays and Sub Pilot-Scale Reactor Performance” was prepared for submission to *Transactions of the ASABE*. This article compares the ability of ATAs and BMPs to predict levels of process inhibition as measured by methane production in sub pilot-scale reactors.

There are three appendices attached which describe additional information relevant to the research performed in this thesis. The first appendix documents the construction of the sub pilot-scale anaerobic digesters. The second appendix gives instruction of on-site monitoring of anaerobic digestion system. The third and final appendix contains items from the educational extension events such as schedules and survey results.

Literature Review

Since the digestion of manure alone offers a relatively low biogas (methane) yield, supplemental materials to increase energy potential have been sought. Extra biogas and/or electricity production and tipping fees can offset large capital costs, making AD more economical (Braun, 2003). With the acceptance of a large variety of

substrates for anaerobic co-digestion comes a new set of challenges, ranging from collection and handling of materials with widely different physical and chemical properties, to process inhibition effects. To solve such problems prior to full-scale implementation, treatability studies are recommended. Studies should be used to establish methane yields, organic loading rates, hydraulic retention times, toxicity issues, ideal mixes, and other parameters relevant to AD design (Wilkie et al., 2004). Some recent studies that have investigated the effects of different co-substrates are listed in Table 1. Each article provides insight on the handling and performance of specific waste streams; however, none of these authors provide a formal procedure or process to analyze multiple substrates for co-digestion or ideal mixture formulation.

Table 1. Literature Review of Recent Co-Substrates

Co-Substrates	Article
brewery wastewater and brewery wastewater solids	Agler et al., 2010
Fresh vegetable waste, precooked food waste, agro-industrial wastewater sludge	Carucci et al., 2005
cheese-making wastewater, poultry breeding wastewater, and olive-oil mill wastewater	Demirer et al., 2001
diluted poultry-manure and olive-oil mill wastewater	Gelegenis et al., 2007
primary sludge, thickened waste activated sludge, and polymer-dewatered fats oils and greases	Kabouris et al., 2009
swine manure and used cooking grease	Lansing et al., 2010
kitchen waste (fried vegetables, starches, rice, meat, etc…) and beef cattle manure	Li et al., 2009
hog waste and poultry waste	Magbanua Jr. et al., 2001
dairy cattle slurry, pig slurry, abattoir wastewater, brewery wastewater, fruit juice wastewater, solid fruit wastes, and dairy wastes (yoghurt and ice cream)	Monou et al., 2009
cattle manure and organic industrial waste (blood from pigs)	Nielsen and Angelidaki, 2008
coarse-cut fodder maize and digester sludge	Raposo et al., 2006
primary sludge, organic fraction of municipal solid waste, and waste activated sludge	Stroot et al., 2001
wheat straw and swine manure	Wang et al., 2009

There are multiple scales of substrate analysis that can be used to identify, characterize, and evaluate materials for co-digestion. Levels range from laboratory scale to bench and pilot-scale for investigation. At the laboratory scale, substrate compounds can be evaluated for carbohydrates, proteins, lipids, and fibers. The subsequent methane potential can then be estimated according to Table 2. (Angelidaki and Ellegaard, 2003).

Table 2. Theoretical Methane Yields of Various Organic Matter Types.

(Based on Angelidaki and Ellegaard, 2003).

Substrate Type	Composition	COD/VS (g COD/g VS)	CH_4 yield (STP L/g VS)[b]	CH_4 yield (STP L/g COD)[b]	CH_4 (%)
Carbohydrate	$(C_6H_{10}O_5)n$	1.19	0.415	0.35	50
Protein[c]	$C_5H_7NO_2$	1.42	0.496	0.35	50
Lipids	$C_{57}H_{104}O_6$	2.9	1.014	0.35	70
Ethanol	C_2H_6O	2.09	0.73	0.35	75
Acetate	$C_2H_4O_2$	1.07	0.373	0.35	50
Propionate	$C_3H_6O_2$	1.51	0.53	0.35	58

[a]Calculations are based on the assumption that all organic matter is solely converted to methane and carbon dioxide.
[b]STP is standard temperature and pressure (0℃ and 1 atm).
[c]Nitrogen is converted to NH_3.

The laboratory methods needed to find the elemental composition of each substrate are time consuming and error prone. Furthermore, this approach does not capture interactions between substrates in a mixture. Because of this, a number of other laboratory techniques were developed to analyze the methane potential of mixed substrates. These include the biochemical methane potential (BMP), dynamic respiration rate (DR_4), and the COD test (Shanmugam and Horan, 2009).

BMPs

The biochemical methane potential (BMP) test provides an indication of the anaerobic degradation and methane formation potential for a substrate or combination

of substrates. It is an experimentally determined value that is typically reported in units of methane volume (mL) per mass (g) of volatile solids (VS). The BMP method was originally described by Owen et al. (1979) as a simple, quick, and inexpensive procedure to monitor relative anaerobic biodegradability. This method was later improved and explained by Speece (2008). The BMP method has been documented in great detail for determining the anaerobic biodegradation potential of a substrate or mixture rather than the methane production potential by ASTM (2008) and ISO (1995) standards. More recent variations of the BMP method have been reported by Moody et al. (2011b) which formalize the inoculum source, nutrient media, and methane measurement through a gas analyzer such as the method used in this thesis. In general, BMPs combine a small amount of substrate with inoculum and a source of micronutrients in an anaerobic environment. Biogas volume and methane production are typically monitored over the course of 30 d. Although the BMP indicates how a waste might anaerobically degrade over time, and offers a better estimate of breakdown efficiency than stoichiometric methods (Shanmugam and Horan, 2009), the high dilutions typical of BMPs mask toxicity issues.

ATAs

ATAs were developed to evaluate the effect of a suspected toxicant on methanogenic activity (Owen et al., 1979). The test is performed using a known rapidly biodegradable substrate and a series of varying levels of the suspected toxicant. Details on the variant of the method used in this work can be found in Moody et al. (2011a). Each assay is seeded with an inoculum, then biogas and methane production are monitored for 5 to 7 d. Since each assay receives identical amounts of inoculum and

standard substrate, any decreases in methane production are attributed to toxicity effects. While ATAs can identify single-component toxicity effects, they do not indicate the nature of the toxicity, nor can they accurately predict the toxicity effects (or lack thereof) from substrate mixtures.

Pilot-Scale Reactors

Relatively small amounts (ca. 1 – 10 g or mL) of substrates or mixtures are used in BMP and ATA tests, which leave them susceptible to error from lack of a representative sample. In addition, BMP and ATA tests are batch processes, as opposed to the continuous flow reactors that characterize most full-scale digesters. Furthermore, as bench-scale systems, they readily mask handling problems that may be crucial to understand. To overcome these weaknesses, without going to full-scale, intermediate or pilot-scale reactors may be used. In this work, we employed plug-flow sub pilot-scale reactors. True plug-flow reactors are continuously fed. An automated feeding system would have been needed to accomplish continuous feeding, and was explored. However, due to varying substrate consistency (i.e. viscosity and total solids) a robust and reliable continuous feed system could not be developed within time and budget constraints. Instead, the reactors received manual semi-batch feedings. The design criteria used were to maintain a 21-d hydraulic retention time (HRT) and a reactor temperature of 35℃ to maintain mesophilic conditions for methanogenic growth. A preliminary design of the sub pilot-scale anaerobic digesters is shown in Figure 1. For more details on the construction process see Appendix A.

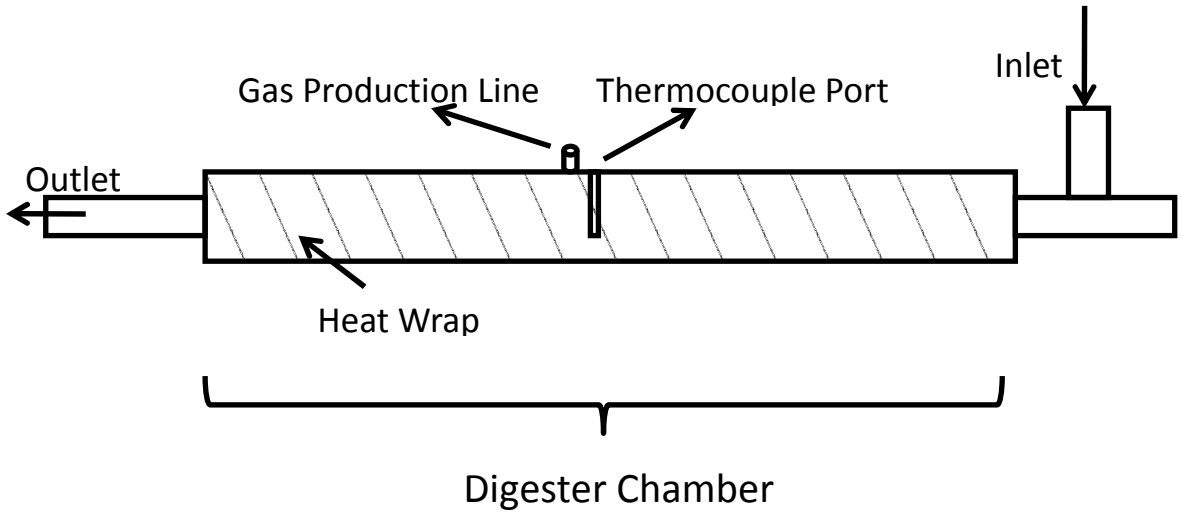

Figure 1. Preliminary design drawing (side view) for sub pilot-scale anaerobic digesters. The components are polyvinyl chloride (PVC) inlet and outlets, high density polyethylene (HDPE) digester chamber, copper thermocouple port, plastic gas production port, and heat trace.

Previous Scale Studies

Cavinato et al. (2010) compared the performance of pilot and full scale completely stirred tank reactors (CSTRs) digesting cattle manure with agro-wastes and energy crops. The pilot-scale reactor was 380 L compared to the full-scale reactor of 1400 m^3 (3700 x larger). They found that the specific gas production was slightly higher in the pilot scale reactor than at full scale, which they felt was due to more efficient mixing. A study performed by Bishop et al. (2009) focused on the ability of BMP methane production from dairy manure to full-scale digester methane production from multiple farms. Their findings indicate that the BMP assays could provide useful information to estimate methane production for dairy manure anaerobic systems as noted by an regression coefficient of 0.53 for the relation between BMP and full-scale methane production in mL biogas per gram volatile solids. Although both studies contribute to knowledge surrounding the comparison between small-scale tests and full-scale reactors, they lack a defined method for selecting co-substrate mixtures and

predicting methane production for digesters of larger scale. This thesis fills the gap between identification of possible anaerobic digestion co-substrates and the use of mixtures in sub pilot-scale digester application to help prevent costly full-scale failure.

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CHAPTER 2. APPROACHES FOR SELECTING ANAEROBIC DIGESTION CO-SUBSTRATES FOR A FULL-SCALE BEEF MANURE DIGESTER USING BIOCHEMICAL METHANE POTENTIALS AND ANAEROBIC TOXICITY ASSAYS

A paper published in *Proceedings of the 2010 International Symposium on Air Quality and Manure Management for Agriculture*

S. T. Sell[1], R. T. Burns[2], D. R. Raman[3], L. B. Moody[4]

Abstract

Design and construction of full-scale anaerobic digesters that co-digest manure with various materials requires analysis of each substrate. Substrate combinations should be analyzed through a scale up procedure in which substrates are characterized, and then evaluated using biochemical methane potential assays (BMPs) and anaerobic toxicity assays (ATAs). The BMPs provide a preliminary indication of the biodegradability of a substrate and of its potential to produce methane via anaerobic digestion, while ATAs determine the degree to which a particular substrate inhibits methane production. Mixture combinations that perform well in BMPs and ATAs should be tested in laboratory-scale anaerobic digesters. Once proven in lab-scale reactors for at least three hydraulic retention times, the best mixture should be tested in a pilot-scale reactor. This paper focuses on the first steps in this process using BMPs and ATAs results to select mixtures for laboratory-scale digester testing. The baseline feedstock

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was beef manure obtained from concrete feedlot pens (open and covered) in eastern Iowa. Various bedding materials were available, including oat hulls, corn stover, and wood shavings. To provide additional energy production, industrial byproducts from cardboard manufacturing, enzyme production, and corn and soybean processing were also potential substrates. Substrates were characterized for TS, VS, COD, pH, alkalinity, and ammonia. Then BMPs were completed on all substrates and ATAs were performed as needed. The results reported here were used to develop mixtures for use in laboratory-scale anaerobic digester testing.

Keywords. Anaerobic Digestion, Biochemical Methane Potential (BMP), Anaerobic Toxicity Assays (ATA), Beef Manure

Introduction

The push towards renewable energy and the reduction of greenhouse gas emissions has prompted some farmers to consider installing anaerobic digestion (AD) systems. With low commercial energy prices in the US, operating such systems on manure alone requires large animal numbers to be economical. This has motivated the co-digestion of animal manure with industrial wastewaters or other sources of biodegradable materials for increased energy production (Braun and Wellinger, 2003). However, full-scale AD reliability has been low due to system design and management challenges (USDA – NRCS, 2007). Design and construction of a full-scale anaerobic digester should be validated by a scale-up procedure. Such a procedure should characterize hydraulic retention time (HRT), organic loading rate, and methane yield (Wilkie et al., 2004). The ideal process begins with laboratory characterization of potential substrates, and then uses biochemical methane potential assays (BMPs) and

anaerobic toxicity assays (ATAs) to examine potential mixtures of substrates (Owen et al., 1979). High performing mixtures should be run in laboratory-scale anaerobic digesters to assess issues that may be masked in BMPs and ATAs. The resulting best mixture should be fed to a pilot-scale system to address materials handling issues (e.g., floating solids, clogging) and to provide data for an economic analysis based on realistic biogas production rates. This paper focuses on the first portion of the scale-up procedure in which laboratory-scale tests results of individual substrates were used to develop mixture ratios for three 100-L, plug-flow laboratory-scale anaerobic digesters.

The BMP is a powerful method of establishing baseline performance data for AD (Speece, 2008; Bishop et al., 2009). While BMPs provide information regarding the methane production of a substrate, they are typically highly diluted and may mask potential substrate toxicity (Moody et al., 2011). To overcome this issue, ATAs may be used. They determine how a particular substrate inhibits methane production by examining methane production from a mixture of a known degradable substrate and the test substrate. However, ATAs are feed-limited batch-loaded systems, and are therefore fundamentally different from typical large-scale anaerobic digesters, which are highly loaded, continuous flow devices. Although critical to early stage design, BMP and ATA results may be misleading when applied directly to full-scale operation due to their lack of information addressing HRT, substrate interaction, and continuous organic loading. Yet, scale-up of AD systems has not been widely reported. This paper provides guidelines for scale-up, and reports on the selection of preliminary substrate combinations based on BMP and ATA work.

Materials and Methods

Manure was obtained directly from confined concrete beef cattle feedlot pens (open and covered) in Eastern Iowa. The manure's estimated age was between 2 – 3 d, and the manure was selected from areas with minimal bedding mixed in. Bedding materials such as oat hulls, corn stover, wood shavings, short fiber cardboard waste, and reed canary grass were collected directly from farm stockpiles and were between 1 – 3 mo. of age. Enzyme production wastewater, food scrap waste, corn processing wastewater liquid, and corn processing wastewater solids were collected after delivery to the farm. Their estimated ages were < 1 d. Soybean processing wastewater was collected directly from the plant's wastewater discharge. Lagoon liquid was collected directly from the on-farm lagoon using a dipper. All samples were stored at 4˚C and were analyzed within one week of collection.

Substrates were characterized for total solids, volatile solids, ammonia, alkalinity, and pH by the Iowa State University Agricultural Waste Management Laboratory. The total solids (TS) and volatile solids (VS) concentrations were measured using standard methods 2540 B and 2540 E, respectively. The pH measurements were taken with an Accumet Basic AB15 Plus pH meter and Accumet 13-620-285 pH probe. The chemical oxygen demand (COD) values were measured using Hach DR/890 Colorimeter Procedures Manual, Method 8000 and vials for COD 0-1500 ppm. Ammonia concentrations were measured using standard methods 4500-NH_3-B Preliminary Distillation Step and 4500-NH_3-C Titrimetric Method with 0.1N HCl as the titrant instead of sulfuric acid. Alkalinity was measured using standard methods 2320 B with 0.1 N HCl as the titrant (Standard Methods, 1995). A BMP assay was performed in triplicate for

each of the substrate using a modified version of the International Standard ISO 11734:1995(E). The ATAs were performed in triplicate at seven dilution ratios on suspect substrates using a modified version of the International Standard ISO 13641-1.

Mixtures were designed to meet criteria including use of all available manure, keeping total solids <15% to facilitate pumping, maintaining pH between 6.5 and 8.2 for microbial ecology, providing high COD concentrations to maximize methane production, and achieving low ammonia to avoid toxicity (Speece, 2008).

Laboratory TS, VS, and COD results were used to calculate the sample size needed for a 250-mL BMP assay bottle. Sample sizes were calculated with a target of 125 mL CH_4 produced during a 30-d period, assuming 70% of COD converted to CH_4, and 395 mL CH_4 /g COD reduced (Speece, 2008). This approach yielded average daily biogas volumes that were in a readily measurable range. The BMP reactors were seeded with an inoculum from a 60-L, mesophilic (35℃), continuously stirred anaerobic reactor that was fed a mixture of high-protein dog food and nutrient medium. The BMP reactors were also seeded with nutrient medium containing supplemental inorganic nutrients and alkalinity (Speece, 2008). Inoculum was added for a 2:1 mass ratio between substrate and inoculum VS. Assay bottles were purged with 70% nitrogen and 30% carbon dioxide gas at ~0.5 L min^{-1} for 5 min. Bottles were then capped with septa and zip tied, and incubated at 35℃ on an orbital s haker at 150 rpm. Biogas production was measured daily by inserting a glass syringe into the septum and allowing the biogas pressure to displace the wetted barrel of the syringe. The volume was recorded, and the biogas was injected into an infrared gas analyzer (NDIR-CH_4 Gasanalyzer, University Kiel, Germany) to obtain the methane content (Bishop et al., 2009). A blank

that included the inoculum source but no substrate was run so that each BMP could be corrected for the methane created by the inoculum source.

Materials of unknown toxicity were analyzed using ATAs. The known degradable substrate was a mixture of nutrient broth, yeast extract, and dextrose (D-glucose) in deionized water. Possible toxicants were combined with the degradable substrate and inoculum in seven mass concentrations (also referred to as % inclusions). The ATAs used the same inoculum and nutrient medium as the BMPs. Known-degradable-substrate controls defined the non-toxic methane production level. The control and all seven dilutions for each substrate were mixed in 250-mL serum bottles and were run in triplicate. Incubation conditions, biogas volume measurement, and methane content measurement were identical as for the BMPs. The methane yield during the linear portion of production was determined for all % inclusions and for the control. Toxicant effects were calculated by taking the ratio of the % inclusion yield to the control. Decreased methane production (inhibition) indicates toxicity, and inhibition generally increases as the ratio of test sample to degradable substrate increases. Higher (or equivalent) methane production indicates a non-toxic substrate.

Results and Discussion

Substrate characteristics are shown in Table 3. Although variations are not shown, liquid samples were generally consistent while solid materials had high variations in some measured variables (e.g., 15 – 30% TS in manure samples). Subsample results listed in Table 3 reflect an average of stockpiles, and we used representative samples for the BMP and ATA assays.

Table 3. Characteristics of Available Substrates.

Substrate	TS (%)	VS (%)	pH	COD (mg/L or mg/g)	Ammonia (mg NH_3-N/L)	Alkalinity (mg $CaCO_3$/L)
Off-Site Co-Substrates						
Soybean Processing Wastewater	0.4	0.3	7.45	7,200*	0	300
Corn Processing Wastewater Liquid	8.3	7.6	4.02	107,600*	260	0
Enzyme Production Wastewater	12.8	11.3	5.05	162,300*	3,330	3,190
Food Scrap Waste	15.8	14.5	4.05	330	2,300	0
Corn Processing Wastewater Solids	18.1	17.5	-	208	-	-
Short Fiber Cardboard Waste	49.0	39.4	-	406	400	7,900
On-Site Materials						
Lagoon Liquid	1.3	0.9	7.06	22,500*	2,900	8,560
Raw Manure	17.0	14.0	6.60	156	1,980	6,000
Bedding Materials						
Reed Canary Grass	84.1	78.4	-	732	-	-
Corn Stover	90.3	84.0	-	870	-	-
Wood Shavings	91.8	91.6	-	170	-	-
Oat Hulls	92.1	87.4	-	750	-	-

*COD reported in mg/L.

Figure 2 summarizes the BMP results on a methane volume per mass VS basis. On this basis, soybean processing wastewater appears to be an ideal source. However, the low VS concentration in this material means that the methane production per total mass of substrate is quite low. To address this, BMP results were also reported on a methane volume per total mass basis (Figure 3). This type of comparison is more meaningful for full-scale application since substrates will be loaded on a mass or volume basis. Figure 3 shows the attractiveness of energy-dense bedding materials.

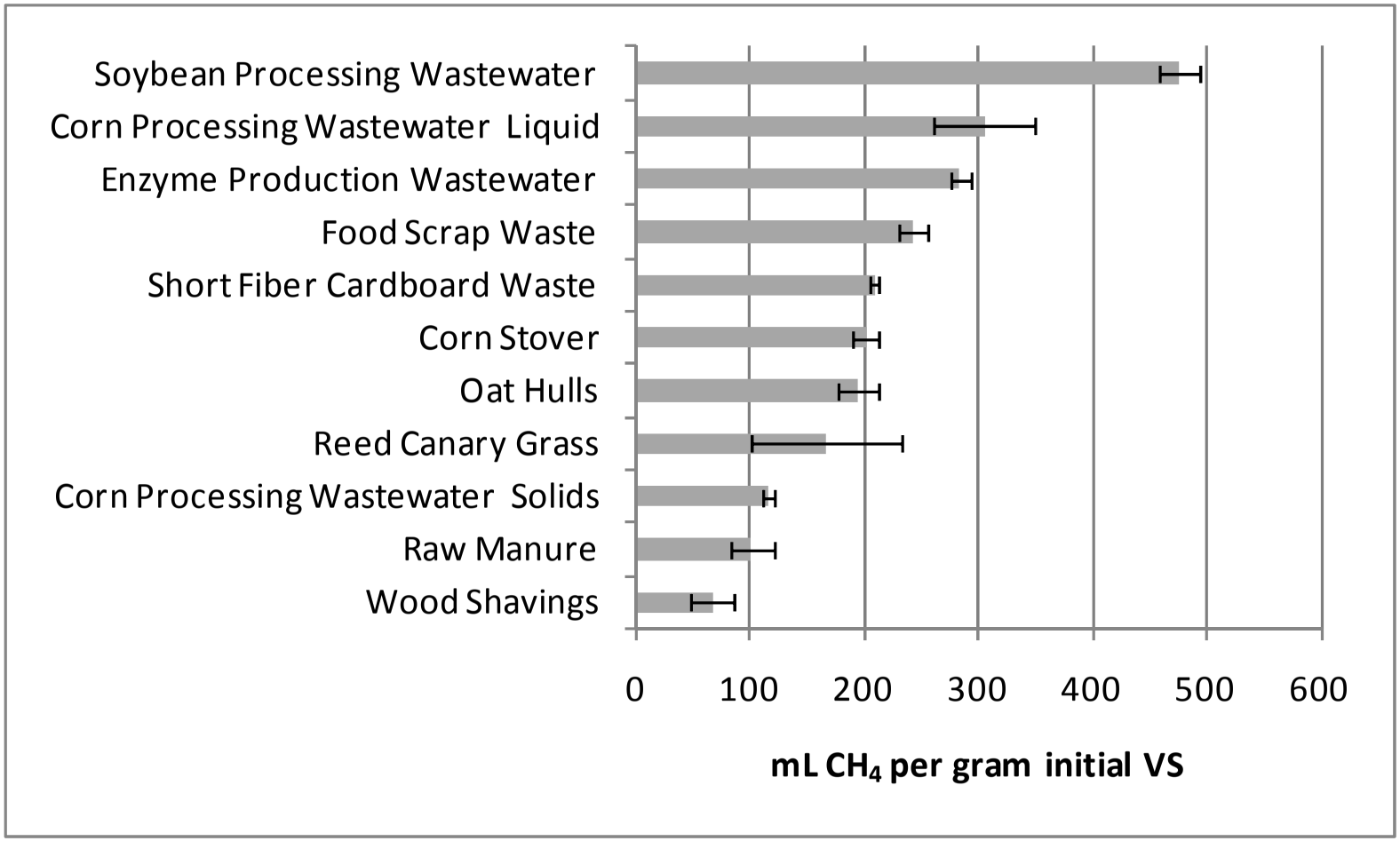

Figure 2. Comparison of substrate BMP results on a per gram of initial volatile solids basis.

Error bars represent one standard deviation of the mean value.

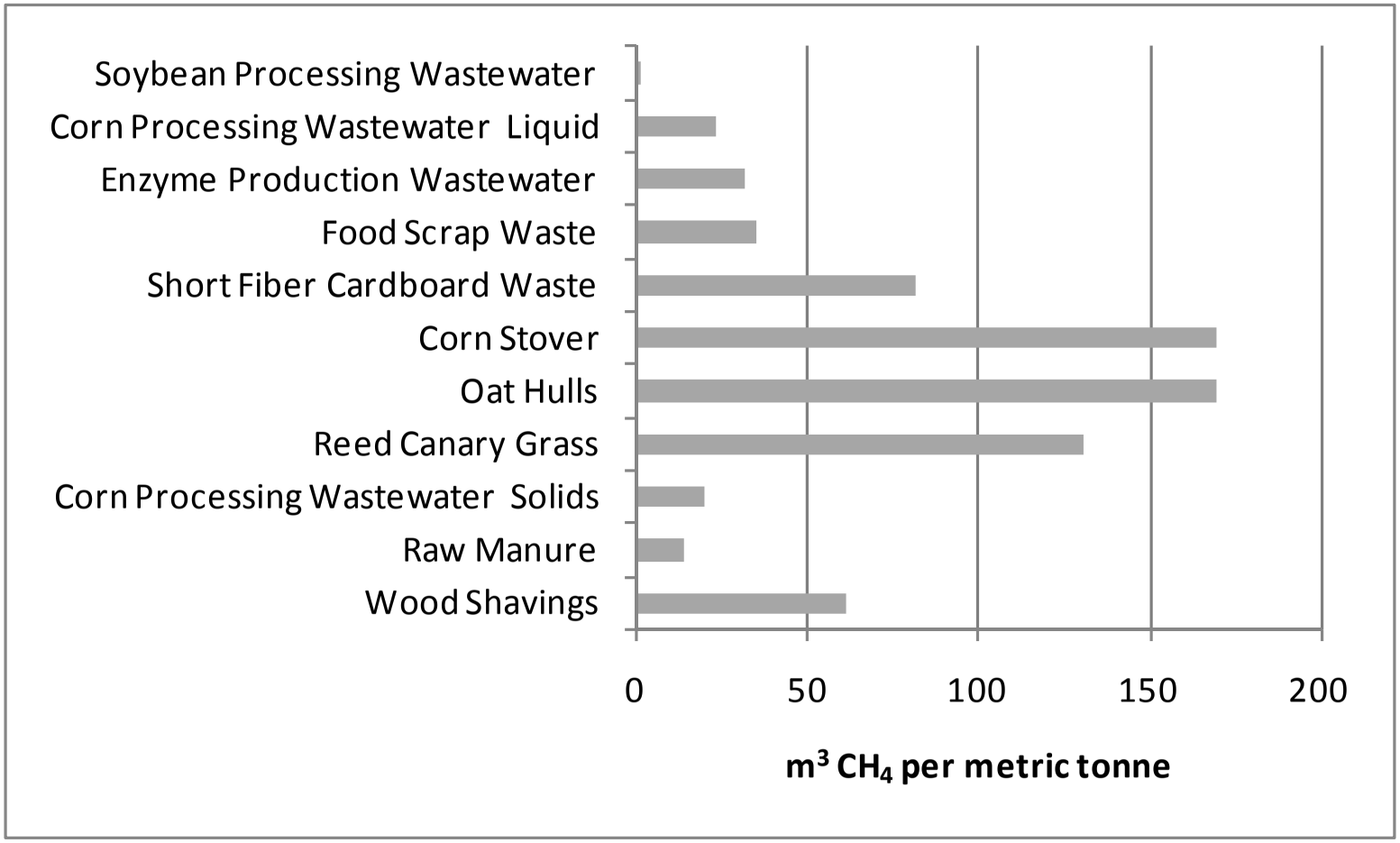

Figure 3. Comparison of substrate BMP results on a cubic meter CH_4 per metric ton basis.

Short fiber cardboard waste, corn processing wastewater, and enzyme production wastewater all showed signs of toxicity in the ATAs, but at varying concentrations. Short fiber cardboard waste was toxic at inclusion rates above 15%. Enzyme production wastewater was toxic at all inclusion rates, perhaps due to its high ammonia levels. Due to the low pH of corn processing wastewater, 50/50 and 23/77 mixtures of manure/corn processing wastewater were examined (results not shown). The manure appeared to act as a buffer to the corn processing waste with negligible inhibition at inclusion rates less than 20%.

Mixture selection was based on material availability and on performance in BMPs and ATAs. Since bedding materials are a portion of the manure, they were not considered as a standalone substrate. Food scraps were available in limited amounts on an irregular basis and were eliminated on that basis. The low COD value and long trucking distance of the soybean processing wastewater caused its elimination, while the enzyme production wastewater was eliminated due to its toxicity. The corn processing wastewater pH was observed to drop rapidly, possibly hindering AD. However, the facility producing the corn processing wastewater was willing to adjust pH prior to delivery. Experiments were run to explore how mixing with manure would buffer this change. If the corn processing wastewater were adjusted to an initial pH of 8.5 with NaOH, a pH above 6.5 could be held for a week with a 10/90 wastewater/manure mixture.

Three mixtures were considered for further testing and evaluation within BMPs. The initial laboratory characterization of these mixtures is shown in Table 4. All mixtures listed in Table 4 use a highly dilute ingredient – either lagoon liquid, screened effluent

liquid, or effluent liquid – to set TS at slightly below 10%. Each of these mixtures will be tested in the plug-flow laboratory-scale anaerobic digester depicted in Figure 4. These 100-L reactors have a design HRT of 21-d and will operate at 35˚C. Mixtures will be allowed to stabilize over the course of three HRTs.

Table 4. Mixture Laboratory Characterization.

Mixture Constituents	TS (%)	VS (%)	pH	COD (mg/L)	Ammonia (mg NH_3-N/L)
Mixture 1					
22% Raw Manure					
15% Short Fiber Cardboard Waste					
16% Corn Processing Wastewater Liquid	9.2	7.2	6.50	80,200	2,150
48% Lagoon Liquid					
Mixture 2					
22% Raw Manure					
13% Short Fiber Cardboard Waste					
16% Corn Processing Wastewater Liquid	9.9	7.7	6.52	86,600	1,480
50% Screened Effluent Liquid					
Mixture 3					
22% Raw Manure					
13% Short Fiber Cardboard Waste					
16% Corn Processing Wastewater Liquid	9.8	7.7	6.53	94,200	1,060
50% Effluent Liquid					

Figure 4. Photo of Sub Pilot-Scale Anaerobic Digesters.

Two of three laboratory-scale 100-L, plug-flow anaerobic digesters. Reactors are aligned with flow counter to each other in this picture. Flow enters at stand pipes and exits through other side. Heat trace is wrapped around each reactor and covered with plastic insulation with a foil backing. Not shown is continuous temperature control is via a PC running LabView and continuous biogas monitoring via inverted tipping-bucket gas meters.

Conclusion

Design and construction of a full-scale anaerobic digester using multiple substrates requires careful selection of substrate mixtures. To select an appropriate mixture, a multi-step procedure is recommended. Initially, substrates are characterized, and then evaluated using BMPs and ATAs. Mixture combinations are then formed using criteria based on the site and data from the BMP and ATA work. Promising mixtures should be further analyzed via BMPs and ATAs, and best performers tested in laboratory-scale anaerobic digesters. This method allows for substrates to be selected and analyzed for any limitations in an anaerobic environment prior to full-scale application, so problems can be minimized. This paper reports results from the first steps of this process, involving characterization of potential substrates, analysis of methane production and possible toxicity, and selection of candidate mixtures. Further research will be performed on candidate mixtures using three 100-L laboratory-scale plug flow reactors. Performance of the mixtures in the 100-L reactors will be compared to that in BMPs to better understand how the BMP mixture results translate in a 500x scale-up (200 mL to 100 L).

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CHAPTER 3. COMPARISON OF METHANE PRODUCTION FROM BENCH- AND SUB PILOT-SCALE ANAEROBIC DIGESTERS

A paper submitted to *Applied Engineering in Agriculture*

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Abstract

Design and construction of full-scale anaerobic digesters that co-digest manure with other substrates, such as food processing wastes, is challenging because of the large number of potential mixtures that can be fed to the digester. In this work we examine the relationship between results from bench-scale methods such as biochemical methane potential assays (BMPs) and sub pilot-scale reactors. The baseline feedstock for this study was beef manure from concrete feedlot pens (open and covered) in eastern Iowa. Additional co-digestion substrates tested were short-fiber cardboard, corn processing wastewater, enzyme processing wastewater and lagoon liquid. Substrates were characterized for TS, VS, COD, pH, alkalinity, and ammonia, after which BMPs were conducted on all substrates. Based on the BMP and ATA results, a mixture was created and evaluated using BMPs and tested in 100-L sub pilot-scale reactors. This study showed that results from BMPs of feedstock co-digestion mixtures accurately estimated the range of methane produced from three 100-L, plug flow reactors.

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Keywords. Anaerobic Digestion, Biochemical Methane Potential (BMP), Co-Digestion, Reactor

Introduction

Co-digestion of animal manure with industrial wastewaters or other sources of biodegradable materials for increased energy production is becoming popular in the U.S. (Braun and Wellinger, 2003). However, full-scale anaerobic digestion (AD) reliability has been low due to system design and management challenges (USDA – NRCS, 2007). Design and construction of a full-scale anaerobic digester should be first validated by less expensive; smaller scale procedures that characterize hydraulic retention time (HRT), organic loading rate (OLR), and methane yield (Wilkie et al., 2004). The ideal process begins with laboratory characterization of potential substrates, and then uses biochemical methane potential assays (BMPs) and anaerobic toxicity assays (ATAs) to examine potential mixtures of substrates (Owen et al., 1979).

The BMP is a powerful method of establishing baseline performance data for AD (Speece, 1996; Bishop et al., 2009). While BMPs provide information regarding the methane production of a substrate, they are typically highly diluted and may mask potential substrate toxicity (Moody et al., 2011a). The ATA was developed to evaluate a substrate's ability to inhibit methane production and therefore determine its potential toxicity. Although critical to early stage design, BMP and ATA results may be misleading when applied directly to full-scale operation due to their lack of information addressing HRT, substrate interaction, and continuous organic loading. However, there have been few publications addressing a proper procedure for AD scale up from substrate identification to full-scale operation. This study aimed to analyze the ability of the BMP

method to predict larger scale anaerobic digestion processes. This paper reports on the performance of individual substrates and a substrate mixture in BMPs and 100-L, plug-flow sub pilot-scale anaerobic digesters.

Materials and Methods

Substrates

Manure was obtained directly from confined concrete finishing beef cattle feedlot pens (open and covered) in eastern Iowa, from a facility where corn stover was the primary bedding material. The diet consisted primarily of corn, distiller's grain, and gluten. At the time of collection, the manure's age was 2 – 3 d, and the manure was selected from areas with minimal bedding mixed in. A wet mill corn processing wastewater and crude glycerin from a soybean & animal lard biodiesel manufacturing facility with were collected within 1 d of delivery to the farm. Cardboard fibers too short for production for a cardboard box manufacturing facility were collected within 5 d of delivery to the farm. Lagoon liquid was collected directly from the on-farm beef manure and separated digester effluent lagoon using a dipper on the side opposite to the influent pipe for maximum lagoon treatment effects. All samples were collected in 20-L buckets, stored at 4˚C, and were analyzed within one week of collection. These substrates were selected out of a list of multiple substrates described by Sell et al. (2010). Selection was based on material availability and on performance in BMPs and ATAs. Industrial wastewaters of choice were not in sufficient quantity to provide all dilution requirements; therefore, on-site water reuse became essential. Since bedding materials were a portion of the manure, they were not considered as a standalone substrate. Some items are not discussed in this paper since they were eliminated from

mixture selection such as food scraps and soybean processing wastewater. Food scraps were available in limited amounts on an irregular basis and were eliminated on that basis. The low COD value and long trucking distance of the soybean processing wastewater caused its elimination, while the enzyme production wastewater was eliminated due to its toxicity. The corn processing wastewater pH was observed to drop rapidly upon sitting, possibly hindering AD. However, the facility producing the corn processing wastewater was willing to adjust pH prior to delivery. Experiments were run to explore how mixing with manure would buffer this change. If the corn processing wastewater were adjusted to an initial pH of 8.5 with NaOH, a pH above 6.5 could be held for at least one week with a 10/90 wastewater/manure mixture. The mixture was designed from these substrates to meet criteria including the use of all available manure, keeping total solids below 15% to facilitate pumping, maintaining pH between 6.5 and 8.2 for microbial ecology, providing high COD concentrations to maximize methane production, and with limited ammonia levels to avoid toxicity (Speece, 1996).

Analytical Methods

Substrates and mixtures were characterized for total solids (TS), volatile solids (VS), ammonia, alkalinity, and pH by the Iowa State University Agricultural Waste Management Laboratory. The TS and VS concentrations were measured using standard methods 2540 B and 2540 E, respectively (Standard Methods, 1995). The pH measurements were taken with an Accumet Basic AB15 Plus pH meter and Accumet 13-620-285 pH probe. The chemical oxygen demand (COD) values were measured using Hach DR/890 Colorimeter Procedures Manual, Method 8000 and vials for COD 0-1500 ppm. Ammonia concentrations were measured using standard methods 4500-

NH_3-B Preliminary Distillation Step and 4500-NH_3-C Titrimetric Method with 0.1-N HCl as the titrant instead of sulfuric acid (Standard Methods, 1995). Alkalinity was measured using standard methods 2320 B with 0.1-N HCl as the titrant (Standard Methods, 1995). A BMP assay was performed in triplicate for each of the individual substrates and mixtures using a modified version of the International Standard ISO 11734:1995(E) per Moody et al. (2011b).

Laboratory TS, VS, and COD results were used to calculate the sample size needed for a 250-mL BMP assay serum bottle (Wheaton Science Products; 250 mL Btl, Serum, Type I Clr, Grad; Millville, New Jersey). Sample sizes were calculated with a target of 125 mL CH_4 produced during a 30-day period, assuming 70% of COD converted to CH_4, and 395 mL CH_4/g COD reduced (Speece, 1996). This approach yielded average daily biogas volumes that were in a readily measurable range. The BMP reactors were seeded with an inoculum from a 60-L, mesophilic (35℃), continuously stirred anaerobic reactor that was fed a mixture of high-protein dog food and nutrient medium (Moody et al. 2011b). The BMP reactors were also seeded with nutrient medium containing supplemental inorganic nutrients and alkalinity (Speece, 1996). Inoculum was added for a 2:1 mass ratio between substrate and inoculum VS. The amounts of each constituent are shown in Table 5.

Table 5. Constituent Breakdown for Individual and Mixture BMPs.

BMP	Substrate Amount	Inoculum (mL)	Basil Media (mL)
Corn Processing Wastewater	9 mL	68	123
Short-Fiber Cardboard Waste	1.8 g	132.2	~66
Enzyme Processing Wastewater	2.7 mL	57.8	139.5
Lagoon Liquid	20 mL	17	163
Raw Manure	2.8 mL	44.7	152.5
Mixture[1]Sample taken at Sub-Pilot Startup	5.5 mL	85	109.5
Mixture[1] Sample taken 3 HRTs into Sub-Pilot Operation	7 mL	100	93

[1]Mixture was composed of (22% raw manure, 14% short-fiber cardboard waste, 16% corn processing wastewater, and 48% lagoon liquid)

Assay bottles were purged with 70% nitrogen and 30% carbon dioxide gas at ~0.5 L min^{-1} for 5 min. Bottles were then capped with septa that were secured with plastic zip ties, and incubated at 35℃ on an orbital shaker at 150 rpm. Biogas production was measured daily by inserting a glass, gas-tight syringe (Micro-Mate Interchangeable Hypodermic Syringe 50cc Lock Tip; Popper & Sons, Inc.; New Hyde Park, New York) into the septum and allowing the biogas pressure to displace the wetted barrel of the syringe. The volume was recorded, and the biogas was injected into an infrared gas analyzer (NDIR-CH_4 Gasanalyzer, University Kiel, Germany) to obtain the methane content (Bishop et al., 2009). A blank that included the inoculum source but no substrate was run so that each BMP could be corrected for the methane created by the inoculum source.

The ATA methodology used at the Iowa State University Agricultural Waste Management Laboratory (ISU AWML) was a modified version of the method performed by Owen et al. (1979) and the International Standard ISO 13641-1 (2003) per Moody et al. (2011a). Aliquots of anaerobic inoculum and an easily degraded standard feedstock were assayed alone (for a fed control) and in combination with a range of eight potential

toxicant inclusion rates. The inoculum source was the same as noted in the BMP method. Once materials were combined in the serum bottles, each bottle was purged with a 70% nitrogen and 30% carbon dioxide gas at ~0.5 L min^{-1} for 5 min. Bottles were then capped with septa and zip tied, and incubated at 35℃ on an orbital shaker at 150 rpm. Biogas production was measured every 24 h over for up to 5 d or until gas production ceased by inserting a glass syringe into the septum and allowing the biogas pressure to displace the wetted barrel of the syringe. The volume was recorded, and the biogas was injected into an infrared gas analyzer (NDIR-CH_4 Gasanalyzer, University Kiel, Germany) to obtain the methane content (Bishop et al., 2009). Results were used to calculate the percent inhibition of methane production for each substrate inclusion rate. Results are reported on a cumulative methane production over a 5 d period or until methane production has ceased as well as on an inclusion verse inhibition basis. In the inclusion verse inhibition display a negative inhibition percentage indicates that a substrate is non-toxic and a positive inhibition indicates signs of toxicity.

Sub pilot-scale Reactors

Sub pilot-scale anaerobic digestion reactors were constructed out of 19.05-mm thick high density polyethylene (HDPE) piping with an inside diameter of 28.45cm. The HDPE pipes were cut to a length of 2.59 m and circular HDPE flanges were extrusion welded on the ends to create the digester chamber. Schedule 80 polyvinyl chloride (PVC) fittings were attached as shown in Figure 5.

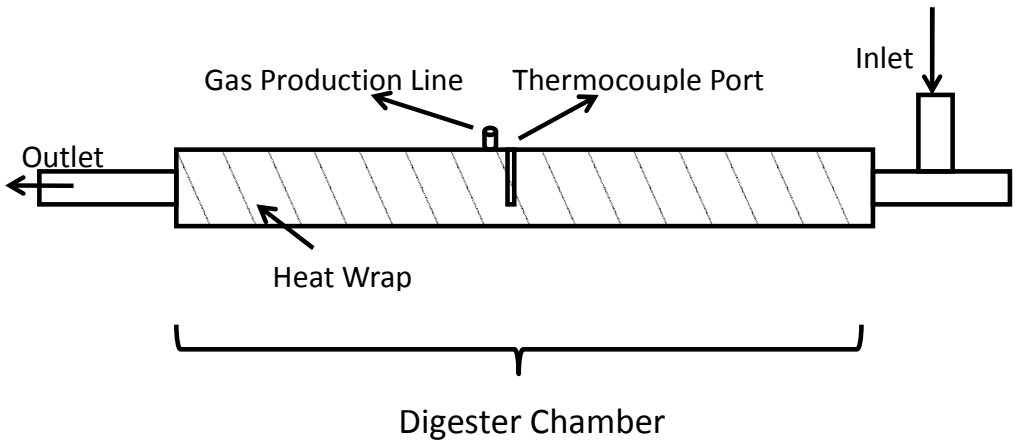

Figure 5. Diagram of sub pilot-scale 100-L, plug-flow anaerobic digester.

Flow enters at stand pipe and exits through other side. Heat trace is wrapped around each reactor and covered with plastic insulation with a foil backing. Not shown is continuous temperature control is via a PC running LabView and continuous biogas monitoring via inverted tipping-bucket gas meters.

Self-regulating heater cable (Nelson Heat Trace; HLT15-J; Tulsa, Oklahoma) was wrapped around the exterior of each digestion tube and connected to a 120 V wall outlet. Plastic bubble wrap insulation with a foil backing was wrapped around the pipe to reduce heat losses from the reactor. Two type-T thermocouples (Omega Engineering, Inc.; EXTT-T-20; Stamford, Connecticut) were placed in the reactor at the axial center, one at the radial cross sectional center of the pipe and the other about 50.8 mm from the internal surface so that both would be submerged in the digestate. The temperature was collected and managed using LabView software (National Instruments Corporation; LabView Version 7.1; Austin, Texas) through personal measurement devices (Measurement Computing Corporation; USB-1208LS, USB-TC; Norton, Massachusetts) connected to a PC. The program was set up in a manner to control the temperature of each reactor at 35°C. A 6.35-mm gas port was insta lled on top of the pipe at the axial center of the digester body and was connected to an inverted tipping-bucket gas meter

submerged in water. Each sub pilot-scale digester had a calibrated tipping-bucket gas meter that recorded gas production amounts using a magnetic reed switch (Digi-Key Corporation; 59065-010-ND, 57065-000-ND; Thief River Falls, Minnesota) via the LabView program. Methane content was determined using 1-L Tedlar bag samples that were measured using an infrared gas analyzer (NDIR-CH_4 Gasanalyzer, University Kiel, Germany). Each digester was started using 100 L of 50/50 water manure slurry that was allowed to reach 35°C for 1 week. Digester 1 was s tarted approximately 3 HRTs prior to digesters 2 and 3 in order to troubleshoot any operation problems before initiation of data collection. Manure was then added following a 21-d HRT until stable gas production was reached. The feedstock was then switched to the mixture and was manually fed in a semi-batch mode (17 L twice per week) that maintained the 21-d HRT.

Results and Discussion

Individual substrate characteristics results are shown in Table 2. Liquid samples were generally consistent, while solid materials had high variations in some measured variables from week to week (e.g., 15 – 30% TS in manure samples). Subsample results listed in Table 6 reflect an average of stockpiles, and we used representative samples for the BMP assays.

Table 6. Characteristics of Selected Substrates.

Substrate	TS (%)	VS (%)	pH	COD (mg/g or *mg/L)	Ammonia (mg NH_3-N/L)	Alkalinity (mg $CaCO_3$/L)	BMP (mL CH_4/g VS)
Off-Site Co-Substrates							
Corn Processing Wastewater	8.3(0.05)	7.6 (0.05)	4.02	107,600(4,500)*	260(10)	0	266(42)
Short-Fiber Cardboard Waste	49.0(0.32)	39.4(0.19)	-	406(61)	400(80)	7,900(370)	208(16)
Enzyme Processing Wastewater	12.8(0.04)	11.3(0.04)	5.05	162,500(9,200)*	3,330(200)	3,190(40)	284(10)
On-Site Materials							
Lagoon Liquid	1.3(0.04)	0.9(0.03)	7.06	22,500(1,250)*	2,900(200)	8,560(400)	356(33)
Raw Manure	17.0(0.50)	14.0(0.81)	6.60	156(28)	1,980(280)	6,000(330)	101(19)

*COD reported in mg/L. Values in parenthesis are standard deviations.

It is important to note that the enzyme processing wastewater appeared to be an ideal dilution liquid based on its BMP results; however, an ATA revealed that even at very low inclusion rates, the wastewater was toxic to the anaerobic consortia. The ATA was determined by comparing methane production from a series of enzyme processing wastewater inclusion rates to a known degradable feedstock (Moody et al. 2011a). It was speculated that the toxicity was due to high ammonia concentrations; therefore, the substrate was dropped as a mixture candidate. A comparison of the selected substrate mixture characteristics is shown in Table 7 and both the average observed values and the predicted values based on a weighted average of the individual component analyses are listed. The observed mixture characteristics represent an average based on influent samples collected weekly for 15 weeks. The differences in the observed and predicted values likely reflect the variable solids in raw manure and short-fiber cardboard waste. However, the COD/VS ratios observed remained very close to the predicted values. (Additional BMP results for these mixed wastes are available in Sell et al., 2010.)

Table 7. Characteristics of the Substrate Mixture.

Characteristics of the substrate mixture, showing actual measurement and the predicted values based a weighted average of the individual components. This mixture was 22% raw manure, 14% short-fiber cardboard waste, 16% corn processing wastewater, and 48% lagoon liquid by volume.

Estimation Type	TS (%)	VS (%)	pH	COD (mg/L)	Ammonia (mg NH_3-N/L)	BMP (mL CH_4/g VS)
Predicted based on individual analyses	11.8	9.6	-	110,600	1,910	202[1]
Average of actual measurements	9.2(2.05)	7.2(1.52)	6.50	80,200(4,930)	2,150(100)	178 (6)[2] 124 (6)[3]

Values in parenthesis are standard deviations.
[1]Predicted on a mass fraction basis from individual results.
[2]Orginial BMP performed during sub pilot-scale startup.
[3] BMP performed during 3rd HRT of sub pilot-scale operation.

Since the mixture BMP was initially performed only on substrates that were collected during the beginning of sub pilot-scale feeding, it did not reflect seasonal changes in substrates. Therefore, another BMP of the mixture was performed from a sample obtained on the 3rd HRT of sub pilot-scale operation. The highest and lowest overall mixture BMP values were multiplied by the influent VS loading, to find a range of possible daily gas productions. These ranges are indicated as dashed horizontal lines on Figure 6. The observed methane flows from the sub pilot-scale anaerobic digester are displayed in Figure 6. Data recording began (0.0 HRT) when the feed was switched from only manure to the mixture discussed in Table 7. Data between 1.43 and 1.62 HRTs were omitted due to their loss during a power outage. The power outage also caused a failure of temperature control, and appears to have led to depressed gas production in the time immediately after the outage (Figure 6).

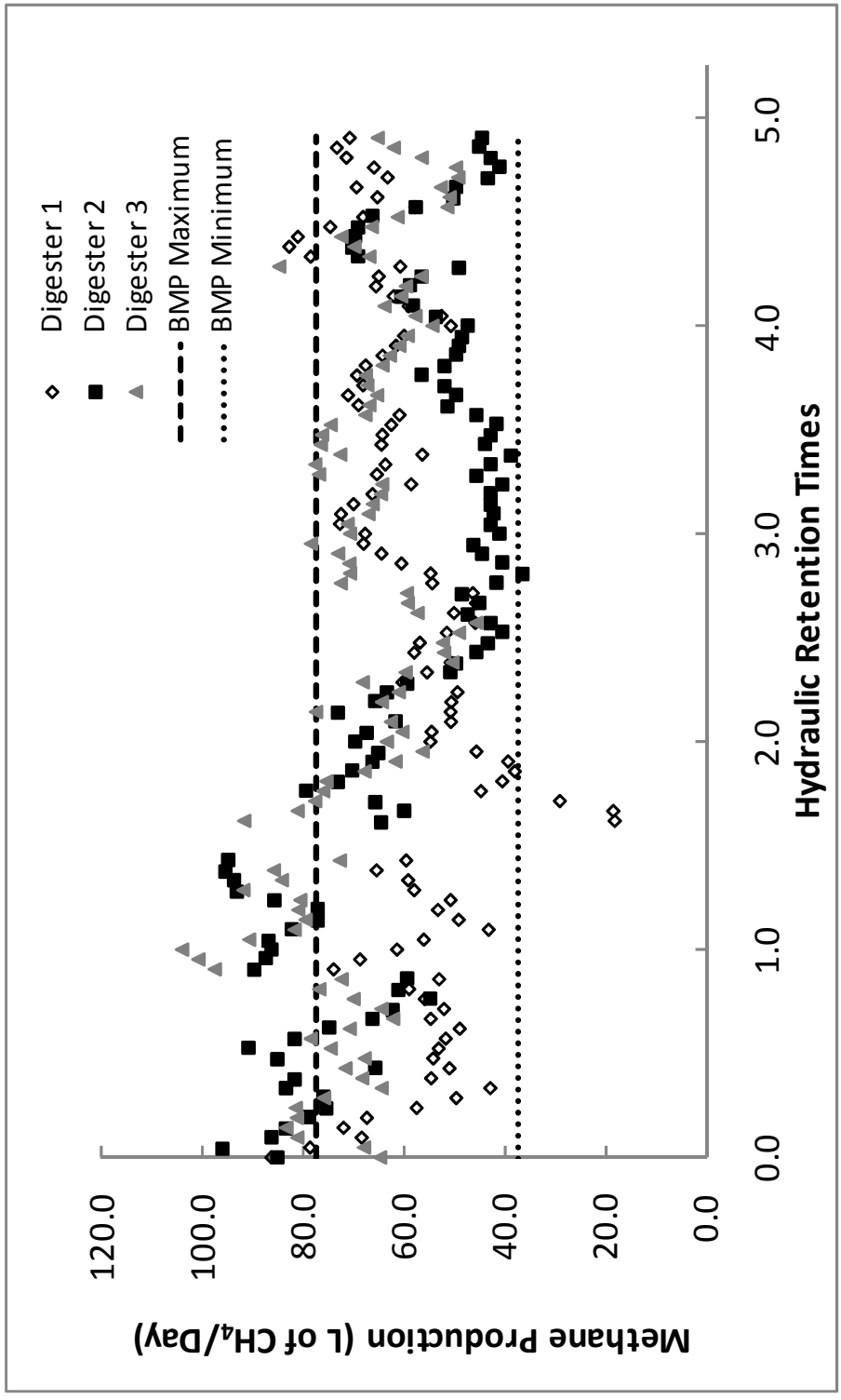

Figure 6. Sub Pilot-Scale Methane Production.

Daily methane production for each pre pilot- scale digester, in comparison to BMP defined gas production values of the mixture.

In Figure 6, the early methane production is above the predicted maximum value (based on the BMPs). This is likely due to degradation of remnants of the inoculum during this time. Digester performance subsequently became more stable in all three reactors, but variations in methane production continued, most likely due to changes in feedstock. The results indicate that mixture BMPs were reasonable predictors of a methane production range for three 100-L plug flow anaerobic digesters. The results also show that BMPs are a snapshot of a real waste, and that temporal variations in the waste can lead to variations in the performance of larger reactors. But equally, the results show that “identical” reactors fed the same waste can have significant variations in gas production. This reflects a combination of the inherent variability of these biological processes and the difficulties in achieving identical conditions in sub pilot-scale reactors fed on mixed wastes.

Conclusion

Determining the best mixture for full-scale anaerobic co-digesters is challenging. This work examined the relationship between results from bench-scale methods such as biochemical methane potential assays (BMPs) and sub pilot-scale reactors. Substrates were characterized for multiple parameters and BMPs were conducted on all substrates. A mixture was designed based on BMP and ATA results, and this mixture was tested in 100-L sub pilot-scale reactors. The BMP maximum and minimum were found to be valid boundaries for the sub-pilot scale ADs after 2 HRTs. Bench-scale methods were helpful in determining larger scale performance while, the sub pilot-scale testing allows materials handling issues (e.g., floating solids, clogging) to be identified,

and provides more robust data for an economic analysis based on realistic biogas production rates.

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CHAPTER 4. DIFFERING EFFECTS OF GLYCERIN ON ANAEROBIC CO-DIGESTION OF MIXED SUBSTRATES IN BENCH-SCALE ASSAYS AND SUB-PILOT-SCALE REACTORS

A paper to be submitted to *Transactions of the ASABE*

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Abstract

Bench-scale methods such as Biochemical Methane Potential (BMP) assays and Anaerobic Toxicity Assays (ATAs) are useful tools in evaluating potential feedstocks for anaerobic digestion. The BMP method provides a preliminary indication of substrate biodegradability and methane production, while the ATAs provide an indication of substrate toxicity to anaerobic microbial consortia. Previous research using small (<20 L) reactors indicated that co-digestion of manures with small amounts of glycerin (ca. 1 – 2 %) can double methane production, but toxicity can result if glycerin exceeds 2% (volumetric basis). This paper investigated the relationship between bench-scale methods (BMPs and ATAs) and sub pilot-scale digester results, using glycerin as a test substrate mixed with a baseline feedstock (beef manure, corn processing wastewater, lagoon liquid, and short-fiber cardboard). The batch-fed, stirred ATAs indicated that glycerin was toxic to methane production at all inclusion levels. The batch-fed, stirred BMPs indicated no significant difference between methane production in the 0.0%to

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4.0% addition levels; however at 8.0% addition, methane production tripled. The continuously fed, non-stirred, plug-flow sub pilot-scale reactors indicated toxicity effects in the 2.0% and 4.0% glycerin mixtures and no difference from the control in the 1.0% glycerin mixture. These results demonstrate the variations in scale performance of glycerin as a co-substrate and identify some serious challenges in extrapolating bench-scale assays to large-scale performance of mixed-waste anaerobic digestion systems.

Introduction

Co-digestion of animal manure with industrial wastewaters or other sources of biodegradable materials for increased energy production has become popular in recent years (Braun and Wellinger, 2003). Substrates of choice are generally high in chemical oxygen demand (COD), low in toxicity, and low in transportation costs. The rise in production of biodiesel in the upper Midwestern US has led to a glut of glycerin (also known as glycerol) in the region, because glycerin is a byproduct of biodiesel production, making it an attractive potential co-substrate since it is readily digestible and easily stored for long periods of time (Robra et al., 2010). The use of glycerin as a co-substrate and its effects on multiple scales of anaerobic digesters has not been widely reported. Instances that have reported on the use of glycerin found that there is a limiting concentration level (Wohlegmut, 2008; Fountoulakis et al., 2010).

The addition of glycerin to hog manure to boost biogas and methane production was studied by Wohlgemut (2008) to determine the ideal ratio of glycerin to hog manure. Four bench-scale completely stirred tank reactors (CSTRs) with a working volume of 3.5 L, and a hydraulic retention time of 17.5 d were employed. The addition of 1% glycerin doubled the methane and biogas production compared to hog manure

without glycerin. At 2% glycerin inclusion, methane and biogas production was the greatest, although a 45-d stabilization period was necessary. The addition of 4% glycerin causes an overloading of COD and failure of the digester (Wohlgemut, 2008). Fountoulakis et al. (2010) focused on the feasibility of co-digesting crude glycerin with sewage sludge in a wastewater treatment plant in both batch and continuous experiments at 35℃. They found that glycerin incre ased biogas yields if the concentration did not exceed 1% (v/v). Above this concentration, organic overloading was thought to cause inhibition of methanogens due to the rapid degradation of glycerin. The addition of long-chain fatty acids have been reported as anaerobic digestion inhibitors, since they cause a lag period in the production of methane from acetate; however, some pretreatment can help reduce chemical oxygen demand and reduce the inhibitory effects (Hanaki et al., 1981). Siles et al. (2010) studied the co-digestion of pretreated glycerin with wastewater in batch laboratory-scale reactors at 35℃. Siles et al. (2010) only reported on experime ntal results of a 15% glycerin – 85% wastewater mixture where they found nearly 100% anaerobic biodegradability to be possible after pre-treatment by acidification and electrocoagulation. They acidified the glycerin with phosphoric acid, and centrifuged the acidified material to recover KOH contamination. In doing so COD of the glycerin was reduced and the possibility of a KOH toxicity problem was eliminated (Ma and Hansen, 2002). Robra et al. (2010) evaluated biogas production on mixtures of 5%, 10%, and 15% glycerin by weight co-digested with cattle slurry. This experiment was carried out in 3 L semi continuous CSTR digesters operated in the mesophilic range. Results show an increase of 9.5%, 14.3%, and 14.6% methane contents respectively for the treatments of 5% glycerin,

10% glycerin, and 15% glycerin compared to a control of cattle slurry without glycerin addition. However, only the 5% and 10% glycerin treatments had statistically higher total biogas yields (normalized per gram volatile solids) compared to the control of cattle slurry only. A failure in the heating system during the 6th week of operation along with high COD, methanol, and KOH concentrations caused the 15% glycerin mixture to have reduced methane production from which it could not recover (Robra et al., 2010). A similar experiment performed by Chen et al. (2008) used CSTR digesters operated at 35℃ using mixtures of 100% glycerin, 60% glycerin/ 40% cattle manure, 45% glycerin/55% cattle manure, and 100% cattle manure on volatile solids (VS) basis. The result was an increase in biogas and methane yields as well as reduction in effluent VS due to greater treatment efficiency for increasing glycerin addition to dairy manure (Chen et al., 2008).

Each of the preceding article results shows a lack of consistency in establishing the correct ratio for glycerin addition as a co-substrate. Our work attempts to use ATAs and BMPs as tools for estimating the methane production of three sub pilot-scale reactors subjected to different glycerin inclusion amounts (1%, 2%, and 4% by volume). The ATAs were developed to evaluate potential substrate toxicity at a bench-scale prior to inclusion in a larger-scale anaerobic system. It was hypothesized that a glycerin ATA would provide information regarding a cutoff point to which glycerin can be added without overloading and causing methane suppression. This information could then be combined with BMP data for different glycerin and co-substrate mixtures to predict the performance of the sub pilot-scale reactors.

Materials and Methods

Substrates

Manure was obtained directly from confined concrete beef cattle feedlot pens (open and covered) in eastern Iowa, from a facility where corn stover was the primary bedding material. At the time of collection, the manure's age was estimated at 2 – 3 d, and the manure was selected from areas with minimal bedding mixed in. We also collected wet mill corn processing wastewater and crude glycerin from a soybean & animal lard biodiesel manufacturing facility; both were collected within 1 d of delivery to the farm. Cardboard fibers too short for production for a cardboard box manufacturing facility were collected within 5 d of delivery to the farm. Lagoon liquid was collected using a dipper on the side opposite to the influent pipe for maximum lagoon treatment effects. The lagoon received beef manure feedlot runoff water and separated digester effluent. All samples were stored at 4˚C and were analyzed within one week of collection. Sell et al. (2010) developed a mixture from these substrates to meet criteria including the use of all available manure, keeping total solids below 15% to facilitate pumping, maintaining pH between 6.5 and 8.2 for microbial ecology, providing high COD concentrations to maximize methane production, and achieving low ammonia to avoid toxicity (Speece, 1996).

Laboratory Methods

Substrates and mixtures were characterized for total solids (TS), volatile solids (VS), ammonia, alkalinity, and pH by the Iowa State University Agricultural Waste Management Laboratory. The TS and VS concentrations were measured using standard methods 2540 B and 2540 E, respectively. The pH measurements were taken

with an Accumet Basic AB15 Plus pH meter and Accumet 13-620-285 pH probe. The chemical oxygen demand (COD) values were measured using Hach DR/890 Colorimeter Procedures Manual, Method 8000 and vials for COD 0-1500 ppm. Ammonia concentrations were measured using standard methods 4500-NH_3-B Preliminary Distillation Step and 4500-NH_3-C Titrimetric Method with 0.1-N HCl as the titrant instead of sulfuric acid. Alkalinity was measured using standard methods 2320 B with 0.1-N HCl as the titrant (Standard Methods, 1995).

BMPs

A BMP assay was performed in triplicate for each of the individual substrates and mixtures using a modified version of the International Standard ISO 11734:1995(E) per Moody et al. (2011b). Laboratory TS, VS, and COD results were used to calculate the sample size needed for a 250-mL BMP assay bottle. Sample sizes were calculated with a target of 125 mL CH_4 produced during a 30-d period, assuming 70% of COD converted to CH_4, and 395 mL CH_4/g COD reduced (Speece, 1996). This approach yielded average daily biogas volumes that were in a readily measurable range. The BMP reactors were seeded with an inoculum from a 60-L, mesophilic (35℃), continuously stirred anaerobic reactor that was fed a mixture of high-protein dog food and nutrient medium (Moody et al. 2011b). The BMP reactors were also seeded with nutrient medium containing supplemental inorganic nutrients and alkalinity (Speece, 1996). Inoculum was added for a 2:1 mass ratio between substrate and inoculum VS. Assay bottles were purged with 70% nitrogen and 30% carbon dioxide gas at ~0.5 L min^{-1} for 5 min. Bottles were then capped with septa and zip tied, and incubated at 35℃ on an orbital shaker at 150 rpm. Biogas production was measured daily by inserting a

glass syringe into the septum and allowing the biogas pressure to displace the wetted barrel of the syringe. The volume was recorded, and the biogas was injected into an infrared gas analyzer (NDIR-CH_4 Gasanalyzer, University Kiel, Germany) to obtain the methane content (Bishop et al., 2009). A blank that included the inoculum source but no substrate was run so that each BMP could be corrected for the methane created by the inoculum source.

ATAs

The ATA methodology used at the Iowa State University Agricultural Waste Management Laboratory (ISU AWML) was a modified version of the method performed by Owen et al. (1979) and the International Standard ISO 13641-1 (2003) per Moody et al. (2011a). Aliquots of anaerobic inoculum and an easily degraded standard feedstock were assayed alone (for a fed control) and in combination with a range of eight potential toxicant inclusion rates. The inoculum source was the same as noted in the BMP method. Once materials were combined in the serum bottles, each bottle was purged with a 70% nitrogen and 30% carbon dioxide gas at ~0.5 L min^{-1} for 5 min. Bottles were then capped with septa and zip tied, and incubated at 35℃ on an orbital shaker at 150 rpm. Biogas production was measured every 24 h over for up to 5 d or until gas production ceased by inserting a glass syringe into the septum and allowing the biogas pressure to displace the wetted barrel of the syringe. The volume was recorded, and the biogas was injected into an infrared gas analyzer (NDIR-CH_4 Gasanalyzer, University Kiel, Germany) to obtain the methane content (Bishop et al., 2009). Results were used to calculate the percent inhibition of methane production for each substrate inclusion rate. Results are reported on a cumulative methane production over a 5 d period or until

methane production has ceased as well as on an inclusion verse inhibition basis. In the inclusion verse inhibition display a negative inhibition percentage indicates that a substrate is non-toxic and a positive inhibition indicates signs of toxicity.

Sub Pilot-Scale Reactors

Sub pilot-scale anaerobic digestion reactors were constructed out of 19.1-mm thick high density polyethylene (HDPE) piping with an inside diameter of 28.5cm. The HDPE pipes were cut to a length of 2.59 m and circular HDPE flanges were extrusion welded on the ends. Schedule 80 polyvinyl chloride (PVC) fittings were attached as shown in Figure 7.

Figure 7. Photo of Sub Pilot-Scale Anaerobic Digesters.

Two of three sub pilot-scale 100-L, plug-flow anaerobic digesters. Reactors are aligned with flow counter to each other in this picture. Flow enters at stand pipes and exits through other side. Heat trace is wrapped around each reactor and covered with plastic bubble wrap insulation with a foil backing. Not shown is continuous temperature control is via a PC running LabView and continuous biogas monitoring via inverted tipping-bucket gas meters.

Self-regulating heater cable (Nelson Heat Trace; HLT15-J; Tulsa, Oklahoma) was wrapped around the exterior of each digestion tube and connected to a 120 V wall outlet. Plastic bubble wrap insulation with a foil backing was wrapped around the pipe to reduce heat losses from the reactor. Two type-T thermocouples were placed in the

reactor at the axial center, one at the radial cross sectional center of the pipe and the other about 50.8 mm from the internal surface so that both would be submerged in the digestate (see Figure 8).

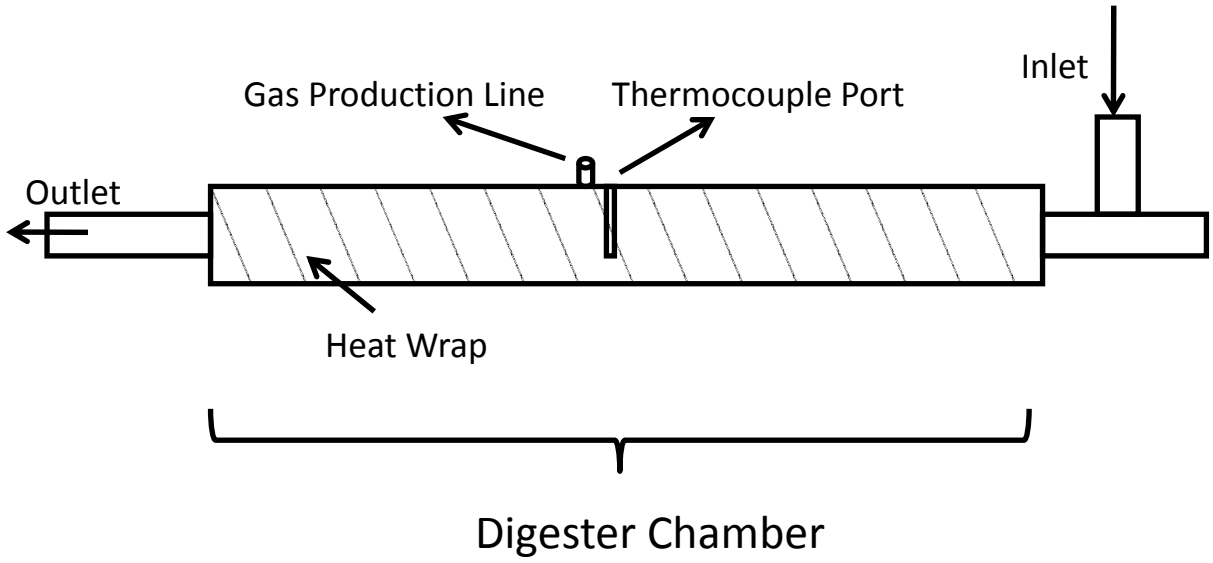

Figure 8. Preliminary design drawing (side view) for sub pilot-scale anaerobic digesters. Side view design drawing for sub pilot-scale anaerobic digesters. This diagram depicts the polyvinyl chloride (PVC) inlet and outlets, high density polyethylene (HDPE) digester chamber, copper thermocouple port, plastic gas production port, and heat trace.

The temperature was collected and managed using LabView software (National Instruments) through a personal measurement device (PMD) connected to a PC. The program was set up in a manner to control the temperature of each reactor at 35°C. A 6.35-mm gas port was installed on top of the pipe at the axial center of the digester body and was connected to an inverted tipping-bucket gas meter submerged in water. Each sub pilot-scale digester had a calibrated tipping-bucket gas meter that recorded gas production amounts using a magnetic reed switch via the LabView program. Methane content was determined using 1-L Tedlar bag samples that were measured using an infrared gas analyzer (NDIR-CH_4 Gasanalyzer, University Kiel, Germany). Prior to experimentation with glycerin as a potential toxicant, each digester was stabilized for

5 hydraulic retention times HRTs on the 22% beef manure, 16% corn processing wastewater, 14% short-fiber cardboard, and 48% lagoon liquid mixture as described by Sell et al. (2011). Each digester was manually fed in a semi-batch mode (twice per week) so that a 21-d HRT was maintained. All mixture components excluding glycerin were mixed every two weeks and stored at 4˚C until ready for feeding. Glycerin was stored in sealed containers held at room temperature (22˚C) and heated to ~35˚C prior to mixing with feedstock using a microwave oven. This was done to increase viscosity and solubility for stirring prior to batch feeding.

Results

Individual substrate characteristics results are shown in Table 8. Liquid samples were generally consistent, while solid materials had high variations in some measured variables from week to week (e.g., 15 – 30% TS in manure samples). Subsample results listed in Table 8 reflect an average of stockpiles, and we used representative samples for the ATA and BMP assays.

Table 8. Characteristics of Selected Substrates.

Substrate	TS (%)	VS (%)	pH	COD (mg/L or mg/g)	Ammonia (mg NH_3-N/L)	Alkalinity (mg $CaCO_3$/L)	BMP (mL CH_4/g substrate)
Off-Site Co-Substrates							
Corn Processing Wastewater	8.3(0.05)	7.6 (0.05)	4.02	107,600(4,500)*	260(10)	0	20.2(3.2)
Short-Fiber Cardboard Waste	49.0(0.32)	39.4(0.19)	-	406(61)	400(80)	7,900(370)	82.0(6.2)
Glycerin	49.7(0.11)	43.6(0.11)	-[1]	>1,000,000	-[2]	-[1]	23.6(8.8)
On-Site Materials							
Lagoon Liquid	1.3(0.04)	0.9(0.03)	7.06	22,500(1,250)*	2,900(200)	8,560(400)	3.2(0.3)
Raw Manure	17.0(0.50)	14.0(0.81)	6.60	156(28)	1,980(280)	6,000(330)	14.2(2.6)

*COD reported in mg/L. Values in parenthesis are standard deviations.
[1]pH and Alkalinity could not be accurately measure since glycerin stuck to pH probe and skewed readings.
[2]Ammonia could not be measured on glycerin due to clogging and sticking of the distiller.

Figure 9 shows the results of the ATA performed on glycerin. It was expected that low glycerin inclusion rates would not cause overloading and would mimic or perhaps exceed the performance of the control. However, at all inclusion rates down to 0.5%, glycerin appeared to suppress methane production. The most likely cause was rapid hydrolysis and acidogenesis occurring from the glycerin and standard feedstock (sugar) which, in turn, dropped pH lower than the methanogens could overcome due to low alkalinity.

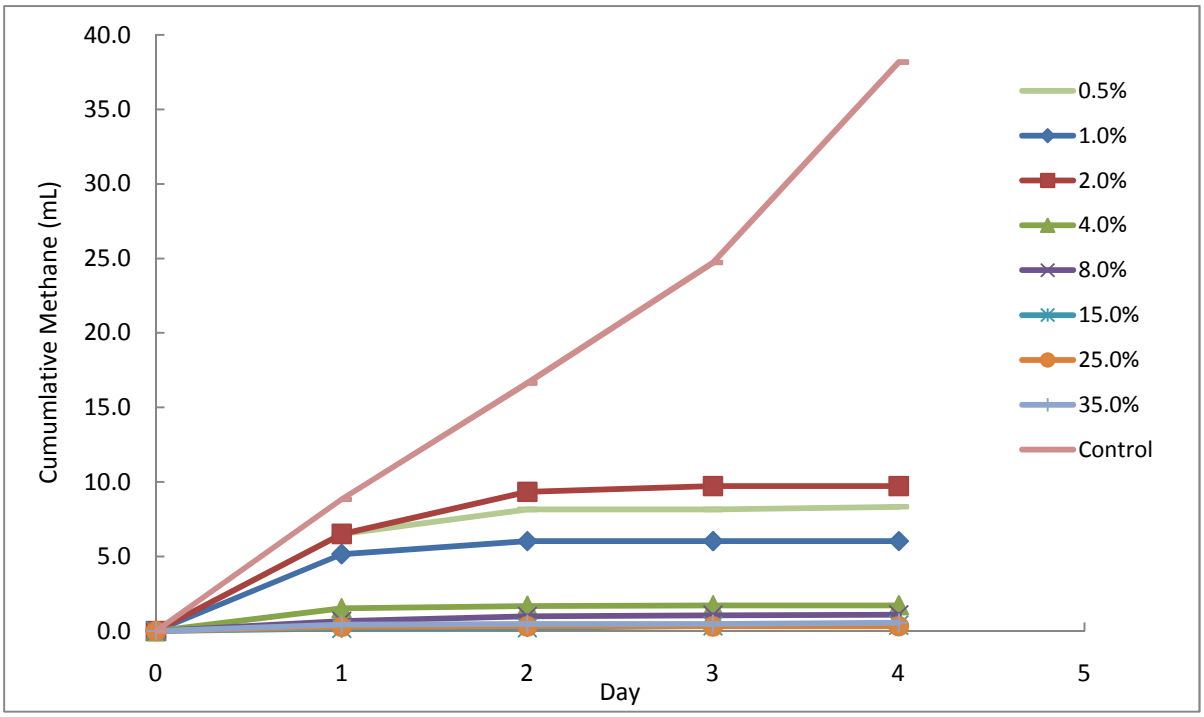

Figure 9. Glycerin ATA.

This ATA was performed at various glycerin inclusion rates as noted in the legend. The control did not contain glycerin and represents the minimum slope for zero methane suppression.

Since the ATA did not provide any information how glycerin would perform when mixed with the baseline feedstock (48% lagoon liquid, 22% beef manure, 16% corn processing waste water, and 14% short fiber cardboard), a series of BMPs were performed with the mixed feedstock plus glycerin. Glycerin was mixed into the baseline

feedstock at inclusion percentages of 0.5%, 1%, 2%, 4%, 8%, 15%, 25%, and 35%. For calculation and comparison purposes 7 mL of each mixture was placed into each 200 mL BMP, thus reducing each glycerin BMP inclusion percentage to 0.0175%, 0.035%, 0.07%, 0.14%, 0.28%, 0.525%, 0.875%, 1.225%. For comparison BMPs containing only the 7 mL baseline feedstock, 7 mL of glycerin (3.5% BMP glycerin inclusion rate), and 3.5 mL glycerin/3.5 mL deionized water (1.75% BMP glycerin inclusion rate) were also performed.

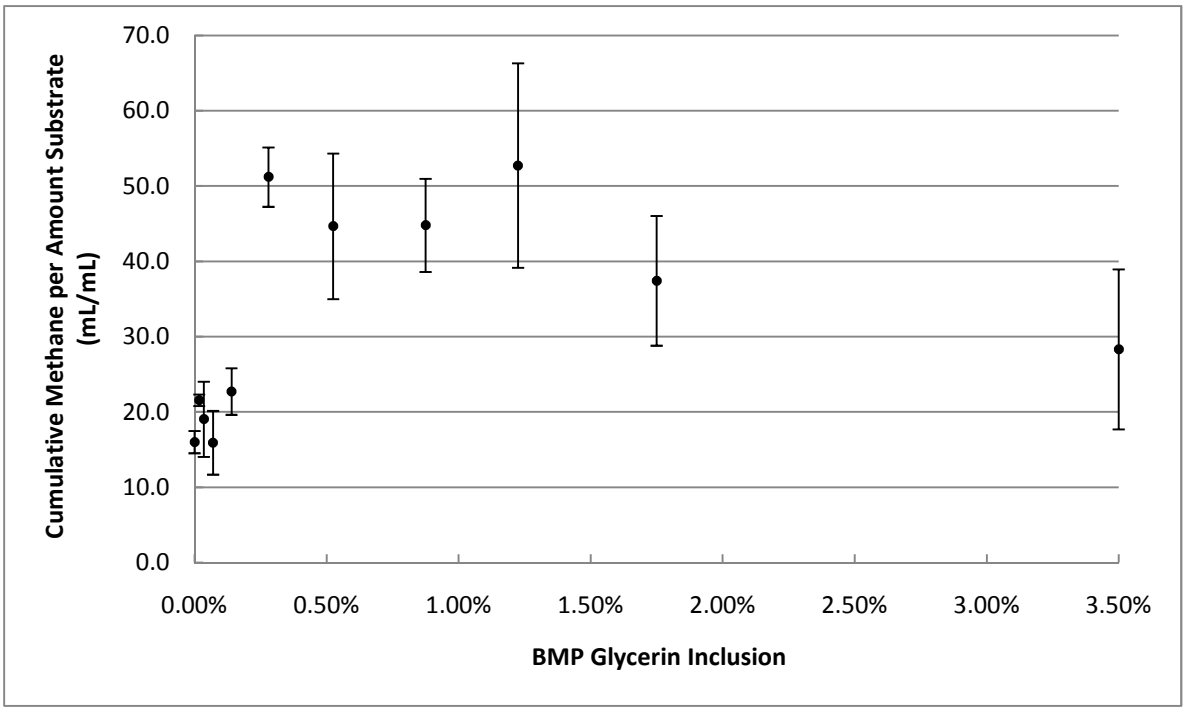

Figure 10. BMP Baseline Feedstock and Glycerin Inclusion Effects on Methane Production.

This plot compares the BMP glycerin inclusion percentage with the cumulative methane produced per amount of substrate loaded on a mL of methane per mL of substrate basis. Error bars depict one standard deviation of the mean.

The BMP results indicate that there were not significant differences for the addition of glycerin to the addition of the baseline feedstock for glycerin mixture inclusion rates of 0.5% to 4% (BMP inclusion rates of 0.0175% to 0.14%). However, upon increasing the

glycerin mixture inclusion rate to 8% (0.28% BMP inclusion rate), the methane production more than tripled. The alkalinity provided by the baseline mixture appears to allow for higher methane production as seen by the lower results for the 3.5 mL glycerin/3.5 mL deionized water (1.75% BMP glycerin inclusion rate) and 7 mL glycerin mixtures (3.5% BMP glycerin inclusion rate).

Figure 11 shows the cumulative daily methane production for each mixture of baseline feedstock and glycerin. Since 7 mL of each mixture was used in each BMP, the comparison of cumulative methane can be made without a correction factor. The first 10 d show rapid methane production for glycerin mixture inclusion rates between 8% and 35% (0.28% to 1.225% BMP inclusion rates) and the methane production is significantly different than the control (baseline feedstock) after the first 5 d. Since the glycerin mixture inclusion rates of 0.5% to 4% (BMP inclusion rates of 0.0175% to 0.14%) do are not significantly different than the control (baseline feedstock), there is a breakpoint between the 4% and 8% glycerin mixture inclusion rates that cause such drastic changes in methane production. The most likely cause is a balance between the amount of carbon or chemical oxygen demand loaded and the alkalinity present to withstand rapid hydrolysis and acidogenesis such that the methanogenic activity was not suppressed. Since these results do not take into account the act of continuous feeding, the sub pilot-scale reactors were performed as a means of comparison.

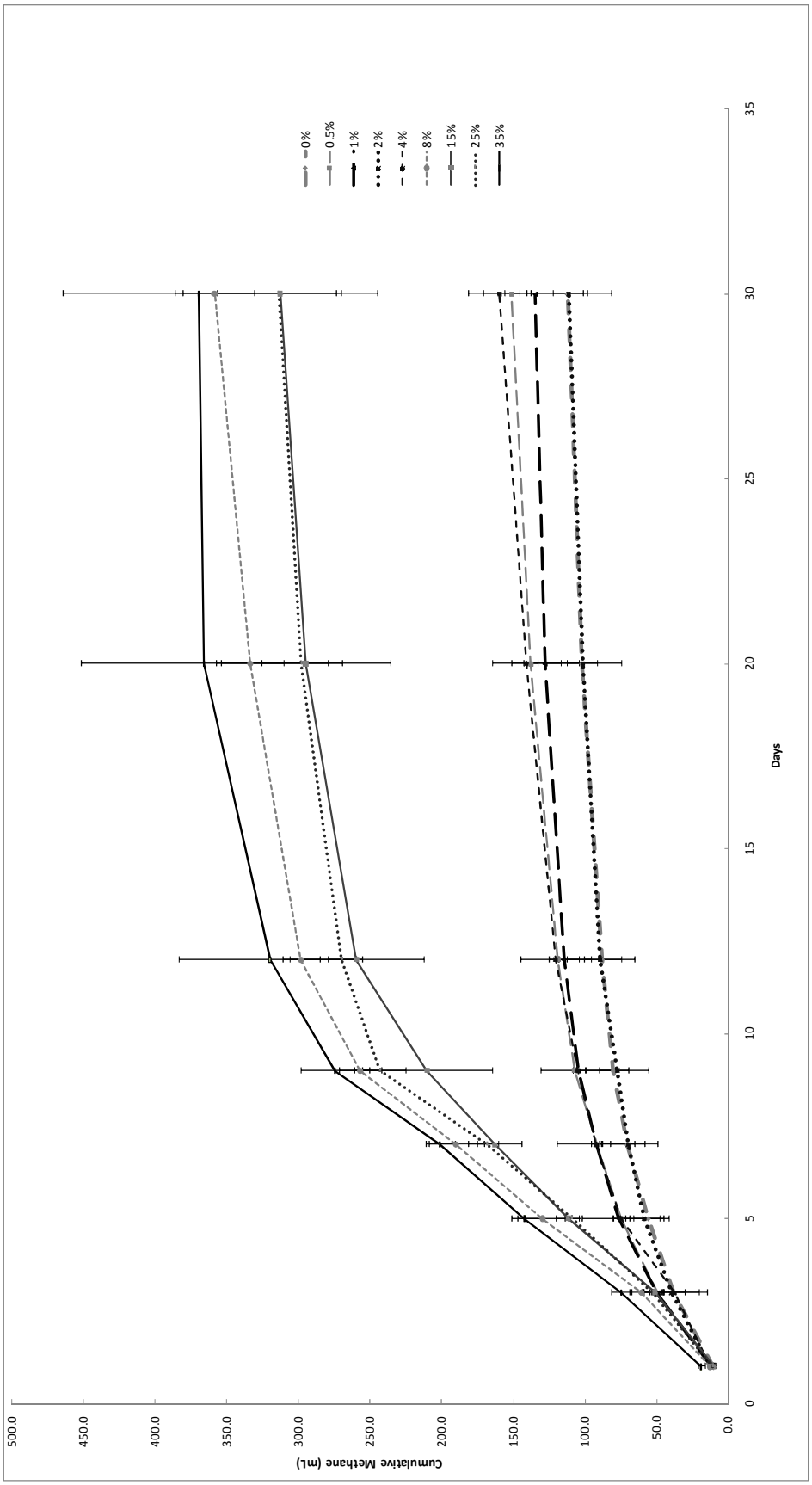

Figure 11. Cumulative Daily Methane Production for Glycerin BMPs.

Daily methane production for BMP mixtures containing 7mL of baseline feedstock mixed with various percentages of glycerin as noted in the legend. Error bars represent one standard deviation of the mean.

The daily methane production results for three sub pilot-scale reactors are shown in Figure 12. Results are shown in L of methane produced per d for three hydraulic retention times prior to the addition of glycerin and for three hydraulic retention times after the addition of glycerin. Prior to the addition of glycerin, sub pilot-scale reactors were operated on the baseline feedstock for over seven hydraulic retention times as noted by Sell et al. (2011). The pre glycerin addition methane production was somewhat sporadic but after the addition of glycerin, each digester separated. At 4% glycerin inclusion, the largest drop in methane production was noticed.

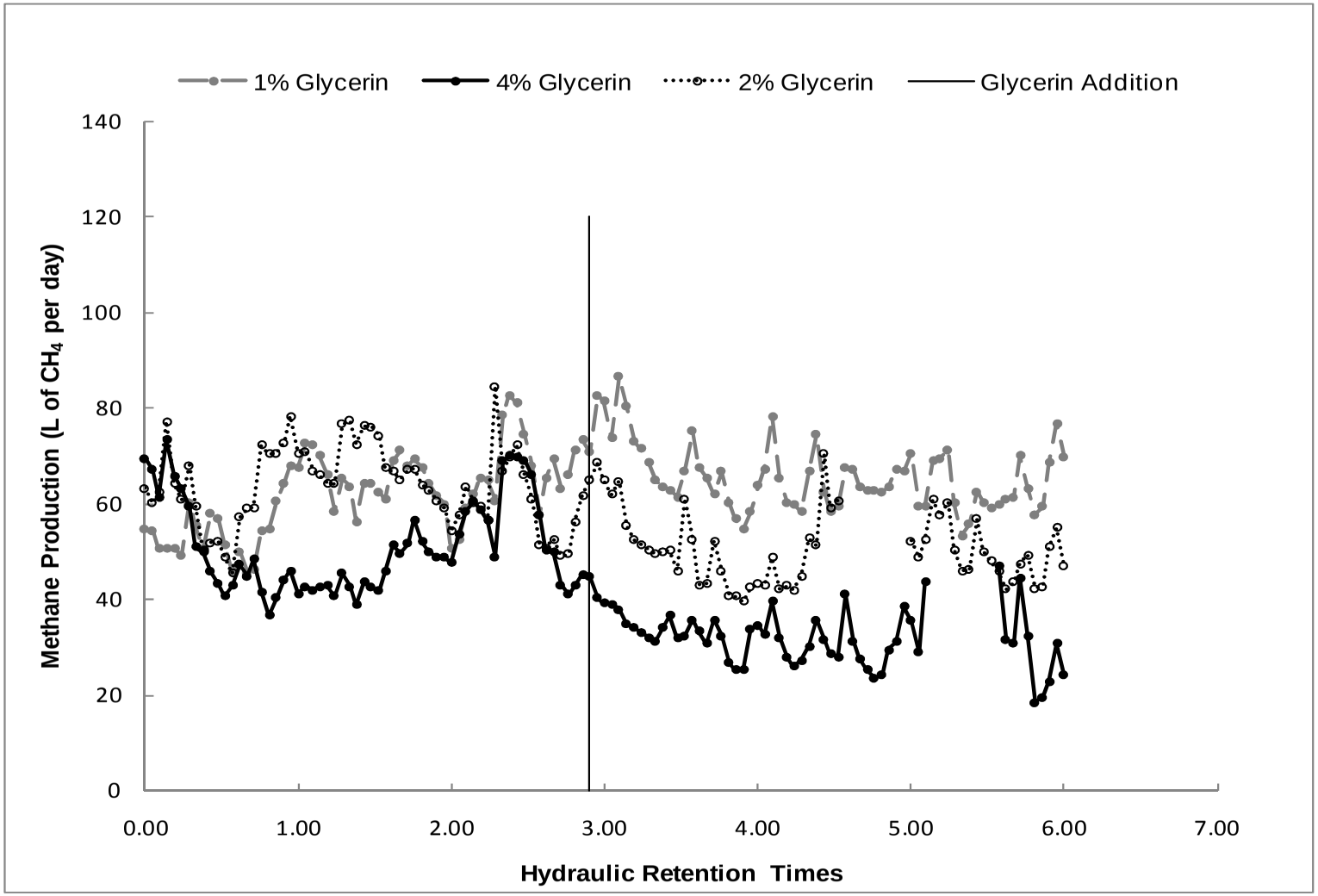

Figure 12. Sub Pilot-Scale Daily Methane Production for Glycerin Mixtures.

Daily methane production results are shown for three hydraulic retention times prior to glycerin addition and three hydraulic retention times with glycerin addition. One hydraulic retention time is equal to 21 d.

Since the information in Figure 12 can be somewhat cloudy in interpreting the effect of glycerin addition on methane production, a bar graph depicting the average control (baseline feedstock) versus each average glycerin methane production is shown below in Figure 13. This graph shows that there were no significant changes during glycerin addition at 1% and 2%; however, at 4% the reduction in methane production is quite significant. There is a visible trend towards lower methane production with an increase in glycerin inclusion percentage.

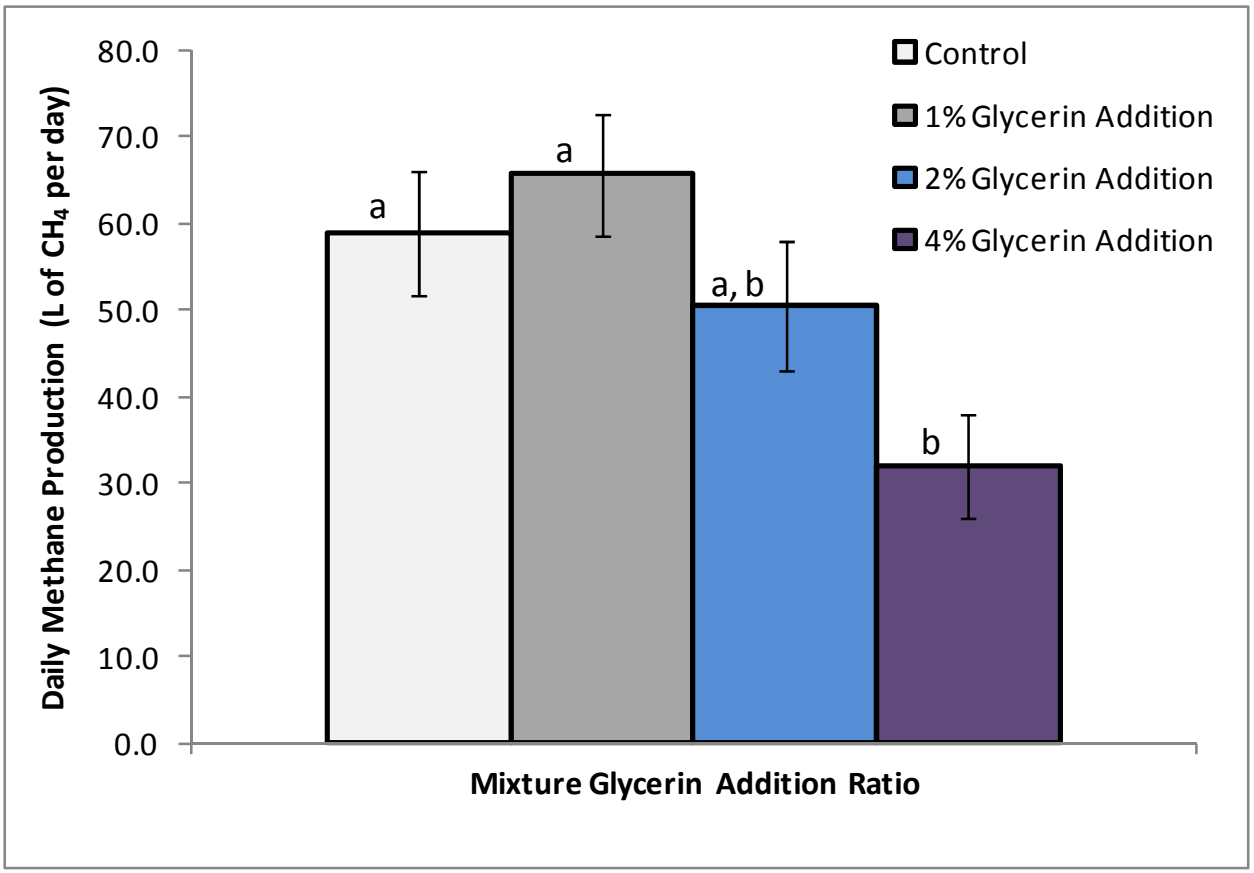

Figure 13. Bar Graph of Average Glycerin Methane Production Compared to Control.

This glycerin addition bars in this graph were formed using the average daily methane production for three hydraulic retention times of glycerin mixture operation. The control (or baseline feedstock) bar was formed using the average methane production for of all three sub pilot-scale reactors during the three hydraulic retention times prior to glycerin addition. Error bars represent one standard deviation of the mean. Similar letters represent treatments not significantly different from each other at a p-value = 0.05.

Although the sub pilot-scale results do not explicitly match those of the BMP or ATA, they do exhibit some similarities. For instance, there does appear to be a toxicity effect from the overloading of glycerin as noted in the sub pilot-scale reactor loaded at 4% glycerin. There also seem to be little no significant changes in low glycerin addition amounts most likely due to the high alkalinity and buffering capacity of the baseline feedstock. It should be noted that the plug flow nature of the sub pilot-scale reactors was susceptible to short circuiting and perhaps settling of solids and more dense glycerin, resulting in poor contact between the glycerin feedstock and the entire microbial population in the digester.

Conclusion

The use of ATAs and BMPs for selection of AD co-substrates and mixtures is a very critical first analysis tool. However, pilot scale studies are very beneficial in analyzing long term performance without the high risk of full-scale failure especially when selecting critical points of substrate addition. This is very important in the digestion of glycerin since it can double or even triple methane production when combined in the right ratio but can be toxic if not combine with proper alkalinity and buffering capacity. This paper demonstrated an ATA that was performed with glycerin inclusion rates of 0.5%, 1.0%, 2.0%, 4.0%, 8.0%, 15%, 25%, and 35% by volume. A set of BMPs was also performed where a baseline mixture was combined with glycerin such that glycerin was 0.0%, 0.5%, 1.0%, 2.0%, 4.0%, 8.0%, 15%, 25%, and 35% of the combined mixture by volume. Control BMPs of 100% glycerin and 50% glycerin/50% DI water by volume were also performed. Three 100-L sub pilot-scale anaerobic digesters were operated at a 21-d hydraulic retention time (HRT) and were each fed in a semi-continuous manner twice weekly (6 loadings per HRT). Each digester was fed a

combination of the mixture selected in paper one with a different amount of glycerin (1%, 2%, 4% by volume). The results of the batch-fed, stirred ATA indicate that glycerin was toxic to methane production at all inclusion levels. The batch-fed, stirred BMP indicated that there was no significant difference between methane production of the 0.0%, 0.5%, 1.0%, 2.0%, and 4.0% mixture combinations; however at 8.0% a triple in methane production was noticed. The continuously fed, non-stirred, plug-flow sub pilot-scale reactors indicated toxicity effects in the 2.0% and 4.0% glycerin mixtures and no difference from the control in the 1.0% glycerin mixture. These results demonstrate the variations in scale performance of glycerin as a co-substrate and identify some serious challenges in extrapolating bench-scale assays to large-scale performance of mixed-waste anaerobic digestion systems. Further study of mixing effects on glycerin inclusion rates is needed to indentify correct loading rates.

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CHAPTER 5. CONCLUSIONS

General Discussion

Anaerobic digestion (AD) is an option for offsetting some dependence on fossil fuels. The challenges of long-term operation of AD systems within the United States are generally related to poor system design, installation, and management. Other factors that prevent the installation of AD systems are low return on investment rate, usually due to low methane generation rates and cheap natural gas prices. In order to supplement methane generation rates, co-digestion and increased organic loading rates has been pursued. In doing so, a plethora of mixture possibilities have arisen. This thesis describes an AD scale-up procedure to assist with selection of materials and mixture performance concerns.

Chapter 2, “Approaches for Selecting Anaerobic Digestion Co-Substrates for a Full-Scale Beef Manure Digester Using Biochemical Methane Potentials and Anaerobic Toxicity Assays,” gave a summary of how BMPs and ATAs were used to narrow down substrates for anaerobic digestion and create mixtures for co-digestion. The ATAs performed helped eliminate enzyme process wastewater as mixture substrate due to its extreme inhibition effects. The BMPs allowed for the comparison and prediction of methane for different mixtures to be tested at sub pilot-scale.

Chapter 3, “Comparison of Methane Production from Bench- and Sub Pilot-Scale Anaerobic Digesters,” compared the performance of a co-digestion mixture in bench-scale BMPs to sub pilot-scale anaerobic reactors. The results showed that methane production stabilization of sub pilot-scale reactors took multiple hydraulic retention times

and were highly dependent on seasonal variation of feedstocks. The BMPs were only able to predict a range for sub pilot-scale operation through the calculation of volatile solids loaded and methane production per volatile solids.

Chapter 4, “Anaerobic Co-Digestion of Mixed Substrates: Relations between Bench-Scale Assays and Sub Pilot-Scale Reactor Performance” compared the ability of ATAs and BMPs to predict levels of process inhibition in sub pilot-scale reactors. An ATA was performed with glycerin inclusion rates of 0.5%, 1.0%, 2.0%, 4.0%, 8.0%, 15%, 25%, and 35% by volume. A set of BMPs was performed where a baseline mixture was combined with glycerin such that glycerin was 0.0%, 0.5%, 1.0%, 2.0%, 4.0%, 8.0%, 15%, 25%, and 35% of the combined mixture by volume. BMPs of 100% glycerin and 50% glycerin/50% DI water by volume were also performed. The three 100-L sub pilot-scale anaerobic digesters were operated at a 21-d hydraulic retention time (HRT) and were each fed in a semi-continuous manner twice weekly (6 loadings per HRT). Each digester was fed a combination of the mixture selected in paper one with a different amount of glycerin (1%, 2%, 4% by volume). The results of the ATA indicate that glycerin was toxic to methane production at all inclusion levels. The BMP indicated that there was no significant difference between methane production of the 0.0%, 0.5%, 1.0%, 2.0%, and 4.0% mixture combinations; however at 8.0% a triple in methane production was noticed. Neither the ATA nor the BMP proved to be an adequate predictor for the sub pilot-scale reactors which saw toxicity effects in the 2.0% and 4.0% glycerin mixtures and no difference from the control in the 1.0% glycerin mixture.

Future Work

Co-digestion of industrial, agricultural, and municipal wastes to produce renewable energy must become a more economical and efficient process for AD success to increase in the United States. Predicting and preventing failure of full-scale operations is possible with pilot and laboratory-scale studies. This thesis demonstrated a procedure that analyzed a variety of possible substrates, selected mixture combinations based on BMP and ATA results, and tested mixture performance in sub pilot-scale reactors. In performing this research, areas of future work became apparent. Some of these critical components include: sub pilot-scale reactor design and configuration, AD kinetics, possible treatment strategies, and economics.

The sub pilot-scale reactors used within this thesis were plug-flow with a 21-d hydraulic retention time and were designed to mimic a full-scale setup. Due to the nature of the mixtures used and the viscosity, it was extremely difficult to prevent clogging of smaller pipes and to prevent short circuiting. Although this gave representation of problems that may arise in a full-scale, plug-flow system, it is recommended that agitation or stirring be used. This will not only keep co-substrates well mixed but also allow for a better comparison to laboratory scale studies.

Future designs should better suit the kinetic breakdown of multiple substrates. Each material will degrade at a slightly different rate; therefore, understanding the kinetics will allow for a better selection of reactor type and the HRT. It would be beneficial to test various HRTs and reactor configurations for glycerin inclusion ratios especially in the 4% to 8% range. This will help determine the most efficient treatment scheme in terms of time and reactor setup. Researching the degradation pathways of

materials will also help identify possibly toxicity issues with substrates and may lead to the timed combination of substrates.

In addition to the breakdown of substrates, identification of methane potential under various pretreatment strategies should be researched. This will help find the ideal strategy for harvesting the maximal amount of energy (methane) from a substrate. Although highly dependent on the cost of the process, a balance between treatment cost and methane production could be formed. Factors of economic importance are a balance between substrate methane production, tipping fees, and operation and maintenance. This will help balance the overall economics of AD systems, which must be studied on an individual scale.

APPENDIX A. CONSTRUCTION OF SUB PILOT-SCALE ANAEROBIC DIGESTERS

The digester chamber material of choice was high density polyethylene (HDPE) due to its durability, strength, and resistance to corrosion or chemical reaction. Each digester tube was built out of 19.05-mm thick HDPE piping with an inside diameter of 28.45 cm. The HDPE pipes were cut to a length of 2.59 m and circular HDPE flanges were extrusion welded on the ends (Figure 14). The HDPE flanges contained a 10.16 cm hole that was matched up with a PVC Flange.

Figure 14. Photos depicting digestion tube end caps.

Left photo shows close up of extrusion weld. Top right photo shows profile view of flanges and extrusion weld. Bottom right photo shows flanges from point of view down the digestion tube.

The combination of length and diameter allowed for the reactor to hold 100 L of substrate above half full, which prevented gas leakage out through the holes to the inlet and outlet. The inlet and outlets were constructed out of schedule 80 PVC with multiple access ports. The inlet featured a stand pipe that allowed substrates to be fed at a head level above the liquid level within the digestion tube (see Figure 15).

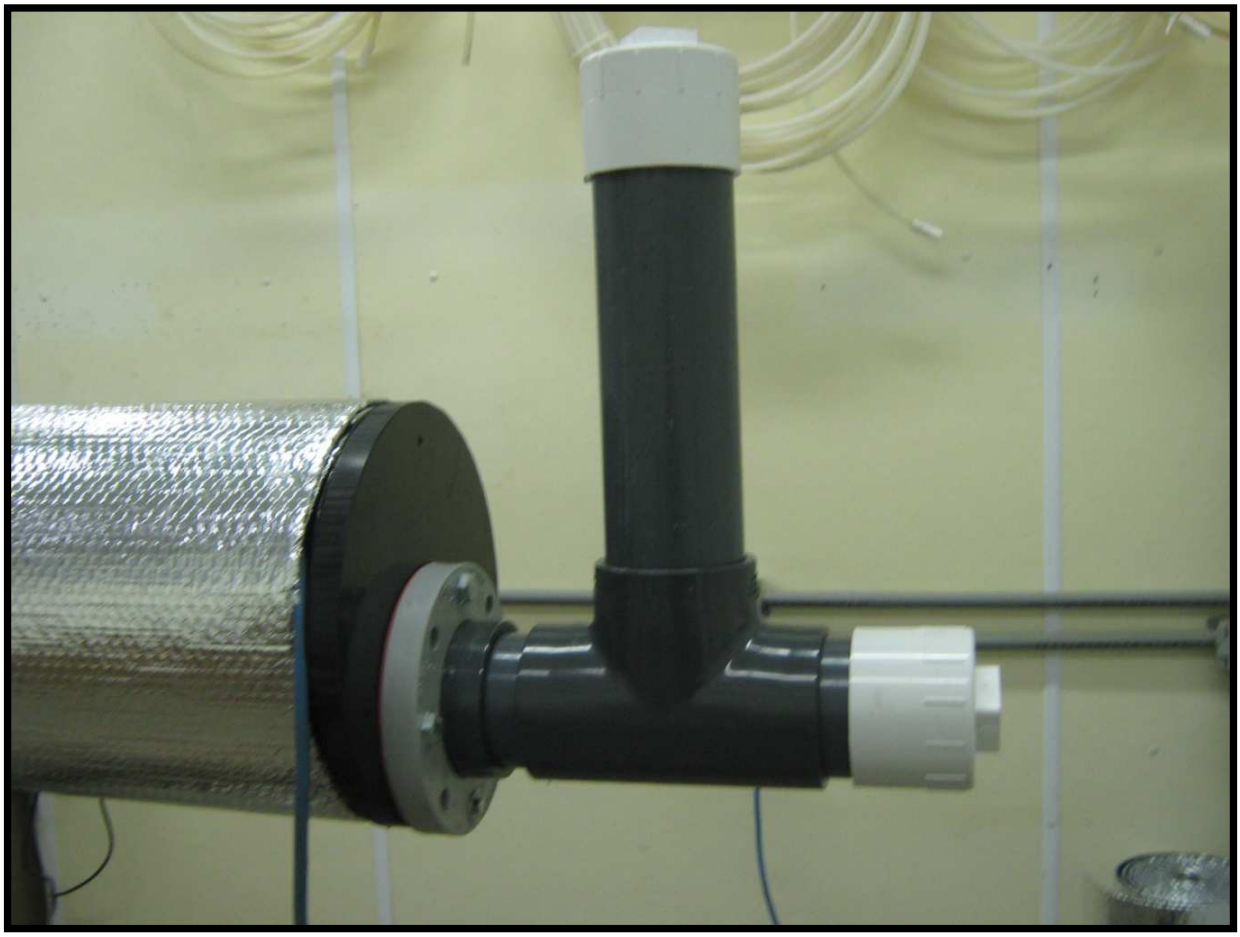

Figure 15. Sub Pilot-Scale Inlet Photo.

Photo of sub pilot-scale anaerobic digester, inlet stand pipe constructed out of schedule 80 PVC.

The outlet of each digestion tube was controlled by a schedule 80 PVC ball valve as shown in Figure 16. This design allows for the controlled release of effluent and directs gas production through the gas port.

Figure 16. Sub Pilot-Scale Outlet Photo.

Photo of sub pilot-scale anaerobic digester outlet ball valve constructed out of schedule 80 PVC.

During semi-batch feeding, the ball valve was opened to allow for removal of digested substrates. Although this released the pressure of the digestion tube, the pressure was quickly recovered upon closure of the ball valve and feeding through the influent stand pipe.

A 6.35-mm gas port was installed on top of the pipe at the axial center of the digestion tube as shown in Figure 17. This port was threaded into the HDPE and sealed with gas tight Teflon tape. Next to the gas port, two type-T thermocouples were placed in the reactor, one at the radial cross sectional center of the pipe and the other about 50.8 mm from the bottom edge. These thermocouples were protected by a sealed copper tube that was threaded into the HDPE using Teflon tape. To provide a more uniform temperature regime, the copper tube was filled with water.

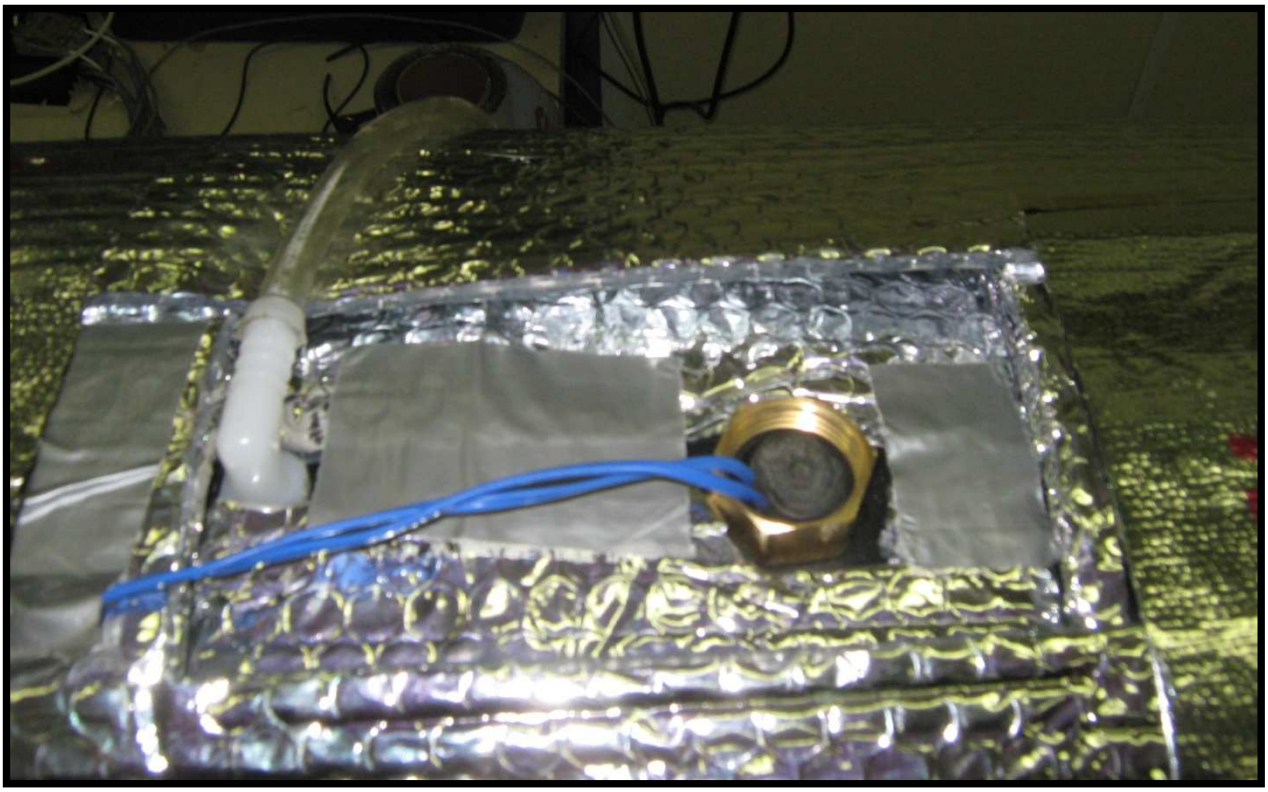

Figure 17. Sub Pilot-Scale Thermocouple Port and Gas Port Photo.

Photo of sub pilot-scale anaerobic digester taken from point of view facing top of axial center. Gas port show in the left of the photo and temperature probe port shown in the right of the photo with two type-T thermocouple wires running into probe. The HDPE tube is surrounded by heat trace and is wrapped with plastic bubble wrap insulation to minimize heat losses.

Digestion tubes were wrapped with Self-regulating heater cable (Nelson Heat Trace; HLT15-J; Tulsa, Oklahoma) around the external surface all the way down the length of

the tube. The heat trace coils were spaced approximately 10.16 cm apart. This heat trace was hooked up to a 120 V wall outlet.

A 6.35 mm diameter Tygon tube was connected to the gas production port and led to an inverted, calibrated tipping-bucket gas meter submerged in water (see Figure 18).

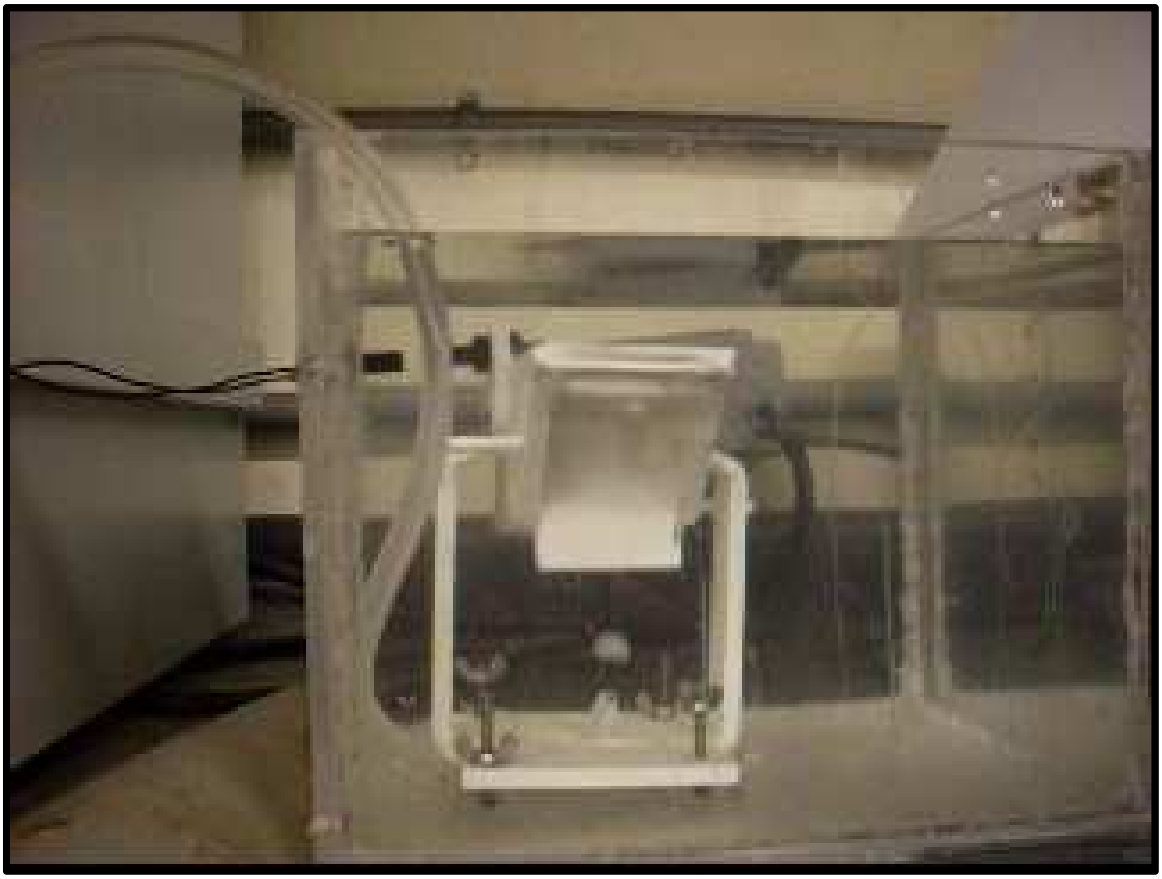

Figure 18. Photo of inverted, calibrated tipping-bucket gas meter submerged in water. Tipping is counted via a magnet reed switch.

Tips from the tipping-bucket are recorded using a magnetic reed switch that is hooked up to a personal measurement device (PMD) that is run by a PC using LabView Software (National Instruments). This LabView Program also takes temperature readings from the two type-T thermocouples and controls the temperature of the digester so that it is at 35℃ by switching the hea t trace on or off.

APPENDIX B. A MANUAL FOR ON-SITE ANAEROBIC DIGESTER PERFORMANCE MONITORING

Introduction

Full-scale anaerobic digestion (AD) processes face numerous operation, maintenance, and management challenges (USDA – NRCS, 2007). Co-digestion of multiple substrates will only increase complications during handling of materials and operation of an AD system. There is a push for full-scale manure digesters within the U.S. to turn towards co-digestion as a means to increase energy production (Braun and Wellinger, 2003). However, farm-owned digesters generally lack access to a sophisticated lab for proper maintenance and monitoring of digester health. This manual provides guidelines for on-site digester performance monitoring of parameters that are critical to the successful operation of an AD system.

How an Anaerobic Digester Functions

The anaerobic digestion process had been used as a means to produce energy rich methane for well over 100 years (Speece, 2008). Further investigation of the chemical and biological processes that control the formation of methane over time has allowed for the adoption of AD systems as a means for waste treatment and biorenewable energy production. A schematic of a typical anaerobic co-digestion process is shown in Figure 19.

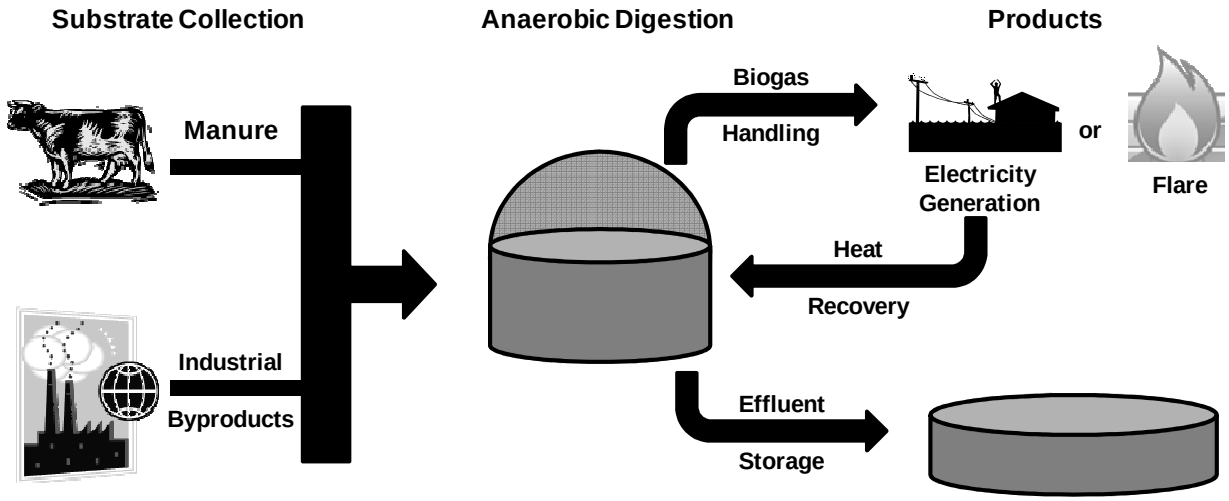

Figure 19. Schematic showing three main steps in anaerobic digestion processes from substrate collection to end products.

Prior to being anaerobically digested, substrates must be identified, collected, mixed and possibly pretreated. Once a proper mixture is selected and placed into an anaerobic (without oxygen) environment, the natural chemical/biological degradation process of digestion begins. The AD process occurs in four reactions steps: hydrolysis, acidogenesis, acetogenesis, and methanogenesis (see Figure 20).

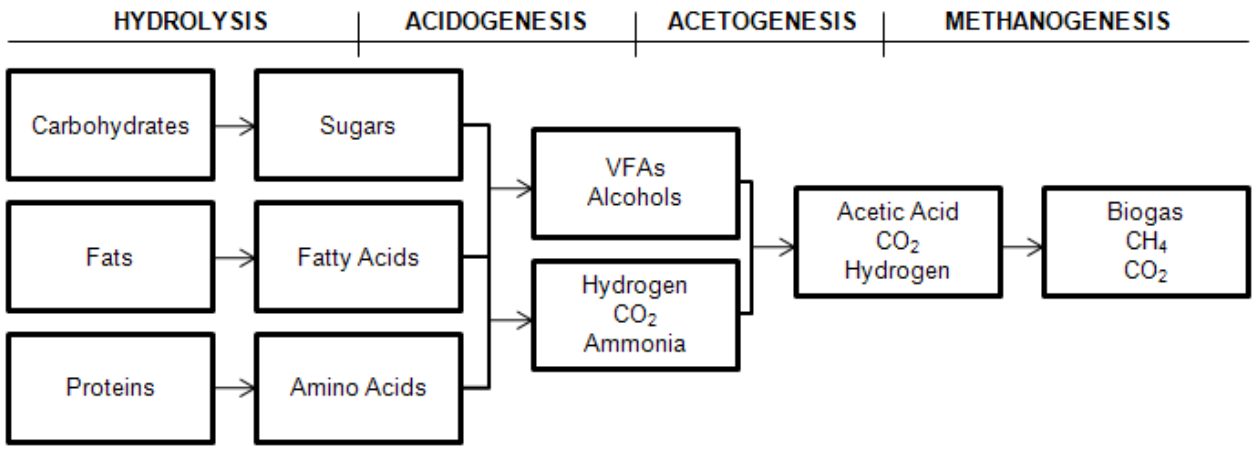

Figure 20. Flow diagram of chemical constituents in an anaerobic digestion process.

Diagram was modified from McNeil, 2005.

AD converts complex organic compounds into simple organic compounds through hydrolysis. These simple organic compounds are converted to long chain fatty acids through acidogenesis and then converted to acetate through acetogenesis. Methanogens then convert acetate (acetic acid) and hydrogen to methane (Speece, 2008).

Key AD Relationships

Hydraulic Retention Time, Volume, and Flow Rate

The term hydraulic retention time (HRT) refers to the average time a single molecule of liquid substrate remains in the anaerobic digester. Although the waste constituents and reactor design play a part, the typical AD system has an HRT upwards of 10-20 d. This is to say that the liquid placed into a reactor will remain there for 10-20 d before exiting the other end (Speece, 2008). The HRT can be calculated using the following equation:

$$\mathrm{HRT} = \frac{\text{Reactor Volume}}{\text{Influent Flowrate}}$$

Equation 1.

Since reactor volume will be a constant, the HRT of a system is dependent on the influent flowrate. In order to maintain a healthy HRT a consistent flowrate or feeding scheme is critical. Increasing the flowrate beyond the recommended HRT of a particular reactor configuration can result in a ‘washout’ or the loss of anaerobic bacteria critical for AD and methane production. Too low of a flowrate and the digester may be starved of nutrients or settling may occur.

Organic Loading Rate, Flow Rate, and Inlet Concentration

The organic loading rate (OLR) of a digester is a measure of the amount and rate of degradable material applied to a reactor. This can be calculated by multiplying the concentration of the organics of the inlet by the flow rate and dividing by the reactor volume as shown in Equation 2.

$$\text{Organic Loading Rate (OLR)} = \frac{\text{Influent Flowrate} \times \text{Influent Concentration}}{\text{Reactor Volume}}$$

Equation 2.

OLR may be expressed as mass of COD per time per volume. Higher OLRs are typically experience with high influent concentration since reactor volume is fixed and an increase in influent flowrate will decrease the HRT of a design system.

Biogas Production and Organic Loading Rate

Biogas production is a result of degradation of organic material. A more concentrated organic material will typically produce biogas at a faster rate than a more diluted organic material. Therefore, a higher OLR is more likely to produce larger quantities of biogas; however, changes in OLR will cause inconsistent gas production. OLR should be maintained by adjusting influent concentration rather than influent flowrate as flowrate adjustments will compromise the design HRT of a system.

Biogas Production and Temperature

Reactor temperature is a critical design parameter that must be maintained in order to achieve the desired AD. There are three general temperature ranges for which AD processes occur. These are psychrophilic (-15℃ to 10℃), mesophilic (25℃ to 40℃), and thermophilic (45℃ to 80℃). The most co mmon is mesophilic zone since is contains the temperature range that mimics the internal temperature of animals. This

allows for less microbiological adjustments to be made during AD. Maintaining a consistent temperature will allow for the most efficient microbes to form; therefore, maximizing the biogas production potential.

Biogas Production and pH

Biogas production is largely dependent on pH of the reactor environment. A range of 6.5 to 8.2 is recommended to satisfy the anaerobic process (Speece, 2008). The first few reactions of AD will cause an increase in acetic acid and a decrease in pH. Neutralization of these acids can cause a reduction in carbon dioxide production which in turn can cause excessive alkalinity. This rise in alkalinity will inhibit methanogenesis and decrease overall biogas production. A balance between alkalinity and pH is critical for maximum biogas (methane) production.

Performance Monitoring Parameters

The parameters noted below are defined for their significance in predicting and monitoring digester health. Although robust at types, measurement and verification of these parameters allows for a looking glass into chemical, biological, a physical performance.

Mixture Constituents

A digester's performance is heavily dependent on the substrates used for digestion. The amount (volume and mass) and type of each constituent loaded into the digester should be recorded. Correlation plots between substrate mixture and methane production can be very helpful in evaluating substrate interactions and poor mixture combinations. Although constant mixture changes at full-scale are not recommended,

they might tend to occur. In order to avoid unsuspected issues at full-scale, it is ideal to have a characteristics map of each individual substrate performed on a lab-scale basis. This provides information such as possible toxicity, biochemical methane potential, pH, etc… Having this performed on each possible substrate allows a mixture prediction spreadsheet can be created for estimation of total solids, methane production, ammonia concentration, etc… Detailed records of loading are a must keep for any data analysis.

Hydraulic Retention Time

Control of the AD system HRT is crucial to stable performance. Recording of substrates loaded daily is the best way to verify that the HRT is being met. If feeding cannot occur in a regular fashion the HRT will continually change and the reactor will most likely not remain stable. Amounts and times should be noted during loading to that a correlation between methane or biogas production and can be made.

Total and Volatile Solids

The total solids content and can be measured using a gravimetric scale and an oven set to 105℃ by following standard methods 254 0 B (Standard Methods). If a 105℃ oven is not available, a rough estimate can b e made by placing a recorded mass of sample in a predried Styrofoam cup and microwaving for 10 minutes at 520 W and letting cool in a desiccator (Dzurec and Baptie, 1989). The mass left over is the total solids mass which can be expressed as a percentage if multiplied by 100 and divided by the initial sample mass. Total solids can be broken down into two categories: volatile solids and fixed solids. Volatile solids are the fraction of total solids that is of organic origin and will be lost during ignition at 550℃. F ixed solids are the ash portion of total solids remaining after ignition at 550℃. Volatile and fixed solids can be measured using

a gravimetric scale and muffle furnace by following standard methods 2540 E (Standard Methods). Volatile solids concentration is a critical component to digester loading since it carries the materials that are capable of anaerobic conversion to biogas. A general rule of thumb is that an increase in volatile solids concentration will reflect an increase in methane production potential. If a muffle furnace is unavailable on-site, each material should be sent to a lab for volatile solids analysis. This will allow an on-site operator to estimate volatile solids content of a mixture by using amounts of substrates loaded and total solids concentrations, assuming that substrates are fairly consistent in composition.

Chemical Oxygen Demand

Although volatile solids are an easy-to-measure parameter, chemical oxygen demand, or COD, is more generally used as the methane prediction value of a substrate. COD is a measure of the oxygen equivalent of the sample's organic matter content that is susceptible to oxidation by a strong chemical oxidant. It has been shown that for every 1 gram of COD reduced during anaerobic digestion, 395 mL (at 35℃ and standard atmospheric pressure) of methane (CH_4) will be generated (Speece, 2008). COD measurement can be somewhat lab intensive; however, companies such as Hach manufacture premeasured COD-ready vials that only require a small sample of the substrate, a heated vial digestion chamber, and a colorimeter or spectrophotometer. If on-site operators do not have access to these types of resources, samples must be sent to a lab for COD analysis. On consistent waste streams, COD concentration will generally correlate with volatile solids concentration, so on-site operators could predict

COD based on volatile solids as long as a relationship between the two for that particular substrate or mix has been developed.

pH

The pH of substrate mixtures loaded into the digester should be recorded for every mixing batch. A pH between 6.5 and 8.2 is necessary for the digester to maintain microbial ecology. pHs outside of this range can become toxic to the system and hinder methane production (Speece, 2008). There a multiple handheld pH meter brands that can be to make measurements. The key to accurate measurements is sufficient sample volume and frequent calibration and cleaning of the pH probe. Trend data containing pH values will also serve as an indicator of methane production changes.

Alkalinity

Low pH values can be problematic in the first stages of an AD system since the formation of volatile fatty acids can overtake the system if there is a lack of alkalinity. Alkalinity is the measure of a solution to neutralize acids and is express as equivalents of carbonate or bicarbonate. Laboratory methods that measure alkalinity involve titration of the sample to a baseline pH value. One such method is given by standard methods 2320 B; however, it involves chemicals that may not be available to on-site operators. If this is the case, samples should be sent to a lab for alkalinity analysis on changes in batch loading. Typically manure systems have high alkalinities so it is not normally an issue, but if alkalinity concentration becomes too low, the system could be susceptible to a drop in pH and a reduction in methane production.

Ammonia

Ammonia concentrations of substrates loaded into an AD system are of importance because high levels will inhibit methane production. While a specific cut-off point is variable between systems, McCarty (1964) showed that 3,000 mg/L measured as NH_4-N is toxic at any pH (Speece, 2008). Other studies by van Velsen 1977 and Parkin and Miller (1982) reported no ammonia toxicity at levels between 5,000 and 8,000 mg/L measured as NH_4-N for systems that were acclimatized over a long term. System operation is dependent on consistency, so ammonia concentrations play a role in tracking trends and could diagnose a problem with a particular feedstock. Measuring ammonia is lab intensive and requires a distiller for most measurement methods. It is recommended that an on-site operator send samples periodically to a lab for analysis.

Temperature

Reactor temperature should be monitored and maintained at the manufacturers recommended setting. The installed temperature probes or thermocouples may not be sufficient in capture data from the entire reactor. It is recommended that the operator have ports installed so that a thermocouple pole can be inserted and readings can be taken from all representative locations within the reactor. This will help identify cold or hot spots so that gas production can be maximized. During steady operation, complete temperature readings can be taken monthly; however, during inconsistent operational periods temperature readings should be taken more frequently.

Solids Depth

Reactor designs can vary significantly, but in most cases on-farm reactors are of the plug flow or longer HRT variety. This gives room for settling issues. Ports installed

within the reactor should be used in combination with a probe to judge the degree of settling within the reactor. Variations in solids should be noted by depth and location, since accumulation can result to digester short circuiting and even complete plugging. During steady operation, these values can be recorded monthly; however, during substrate changes or inconsistent periods, frequent monitoring is a must.

Biogas Production

Biogas production of the AD system should be monitored where biogas leaves the digester, and after any type of biogas upgrading equipment (e.g. moisture or H_2S removal). Biogas flow rates should be monitored in actual volume per time such as cubic feet per minute (acfm) or as standard volume per time such as cubic feet per minute (scfm) as long as temperature and pressure are recorded. A continuously recorded system display is critical to analyzing AD performance, since fluctuations can be view instantaneously and correlated with substrate loading or other system parameters.

Methane Content

Naturally, the methane content of an anaerobic digestion process will fluctuate slightly but over long periods of time the content should remain stable or above 60% for most farm digestion processes. Sudden declines in methane content are strong indicators of digestion problems (i.e. change in digester temperature, substrate change, toxicity issue…). However, a drop in methane content is usually a sign that other monitoring was not being performed adequately since methane production would be the last component to be affected by a problem. Nonetheless, methane content is an important parameter to measure since it will affect engine performance. There are

numerous expensive instruments to measure methane that can be installed directly into the gas line such as infrared analyzers. Another possible way to measure methane content is to back-calculate through CO_2 measurement. This relies on the assumption that biogas is comprised mainly of CO_2 and methane with other gasses being <1%. Cheaper alternatives to infrared analyzers can be used such as a Fyrite. This is a simple handheld device that can be used quickly to obtain a methane content by back-calculation.

Hydrogen Sulfide Content

Hydrogen sulfide is a poisonous gas that is produced during the anaerobic digestion process that has the odor of rotten eggs. Hydrogen sulfide can lead to the corrosion and pitting on metal surfaces. It is this reason that hydrogen sulfide is typically removed from biogas through a scrubber. Monitoring of hydrogen sulfide becomes critical in evaluating the performance of a scrubber system or the degree to which metal surfaces are being exposed to a corrosive environment. Hydrogen sulfide content measurement is necessary for adjusting engine performance and evaluating gas safety levels. Measurements can be performed continuous with an instrument analyzer or periodically with sorbent tubes.

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APPENDIX C. A SUMMARY OF ANAEROBIC DIGESTION EDUCATIONAL EXTENSION EVENTS

In order to fulfill additional requirements of the grant that funded this research project, educational extension events were performed to extend knowledge gained during experimentation and literature review to the public. The first of these events occurred in February 2010 and contained the set of presentations listed in Figure 21. This conference was entitled On-Farm Anaerobic Digestion: Considering the Options and was directed towards producers who would likely install an anaerobic digestion system. The topics covered ranged from whether a farm could support an anaerobic digestion system to digester designs and operations. The second page of the flyer, shown in Figure 22, shows a map and signup information. The conference concluded with a tour of a full-scale, mixed plug-flow anaerobic digester. Details of the facility are shown in Figure 23.

A second educational conference was held on October 27, 2010, with the title of Anaerobic Digestion Part 2: Light at the end of the Tunnel or a Train? The goal of this conference was to establish the midway point of the experiments and to focus in on what are the key parameters of operating a full-scale digester with multiple substrates. The schedule of presentations for this conference is shown in Figure 24 and a map of the location as well as contact information is shown in Figure 25. In order to evaluate the quality of the presentations and their impact on the audience's ability to learn and capture relevant information, an anonymous online survey was used. The results for the survey are shown in Figure 26, Figure 27, and Figure 28. The survey garnished nine responses from an attendance of approximately 40 people. This does not represent a

significant number of attendees; however, the feedback that was provided could be used towards future education conferences in terms of setup, topic selection, and presentation style. The main takeaway points from the survey were

- There is room for improvement of conferences to meet audience's needs
- Audience members were most interested on full-scale anaerobic digester operation updates and optimum substrate mixture combinations
- Equipment manufacturer presentations were of little value to the audience
- Topics of interest for future sessions include: waste stream methane production, operation of pilot-scale reactors, daily operation of full-scale digester, effluent fertilizer quality, electricity generation and payback period
- The venue was appropriate and audience members are looking forward to more information.

Amana Farms Renewable Energy Center & Iowa State University Extension Educational Outreach Program

On-Farm Anaerobic Digestion: Considering the Options

Cost: $25 Register On-site

Date: **Tuesday, Feb. 16, 2010 (*new date due to weather cancellation*)**
Location: **Iowa Theatre Artists Co., 4709 220th Trail, Amana, IA**
Time: **Program from 9:00 a.m. - Noon**
Digester Tour Available after Lunch on Own at 1:30 p.m.

8:30	Coffee, Juice, & Pastries
9:00 - 9:15	Introduction & Welcome Amana Digester Goals/Objectives John Peterson, Amana Farms
9:15 - 9:45	Is a Manure Digester Feasible for your Farm: Factors to Consider Robert Burns, Iowa State University
9:45 - 10:15	Manure Digester Opportunities in Iowa - Allan Goldberg, Iowa DNR
10:15 - 10:45	Generating & Marketing Carbon Credits - Dave Miller, AgraGate Climate Credits
10:45 - 11:15	Amana Farms Digester Design & Installation, Steve Dvorak, GHD, Inc.
11:15 - Noon	Amana Farms Manure Digester Experience - John McGrath & Terry Hershberger, Amana Farms
Noon - 1:30	Lunch - On Your Own
1:30 - 2:30	Amana Farms Digester Tour

For Additional Information, contact:
Lara Moody, lmoody@iastate.edu, 515-294-7355
John McGrath, jmcgrath@amanas.net, 319-622-7557

http://www.abe.iastate.edu/wastemgmt/amana-program.html

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IOWA STATE UNIVERSITY Amana Farms

Figure 21. February 16, 2010 Educational Conference Flyer Page One.

Amana Farms Renewable Energy Center & Iowa State University Extension Educational Outreach Program

REGISTRATION FORM - SUBMITT WITH PAYMENT UPON ARRIVAL

NAME:_____

ORGANIZATION:_____

STREET ADDRESS:_____

CITY, STATE, ZIP:_____

EMAIL:_____

Would you like to be contacted with information about future Amana Farms Renewable Energy Center Educational Outreach Programs? ____Yes ____No

Please complete the registration form and submit it on site with the $25 registration fee the day of the event (cash and checks will be accepted).

Figure 22. February 16, 2010 Educational Conference Flyer Page Two.

Digester Design

Type
GHD, Inc. Mixed Plug-Flow

Digester Design Details
- Sized for Manure from 4,000 Beef Cattle and Off-site Waste Streams
- Approximate Volume of 1.67 Million Gallons
- 21-Day Hydraulic Retention Time
- Temperature Maintained at 100°F by Hot Water Waste Heat
- Handles Medium to High Total Solids (<15%)
- Biogas is Treated for Moisture Removal

Digester Feedstocks
- Beef Manure
- Bedding Materials
- Cardboard
- Corn Processing By-Product
- Food Waste

Farm Facts

Current Herd Size
Approximately 2,300 Beef Cattle

Manure Collection Method
Tractor Scraped from Concrete Pens

Post-Digestion Fiber Recovery
Screw-Press Solids Separators
- Three Bauer Model 855 Separators

Renewable Energy Use

Startup Date
Operation began on October 1, 2008

Independent Utility
Energy production is Independently Owned and Operated by the Amana Society Service Company

Installed Generation Capacity
2.8 Megawatt
- Four 710 KW Guascor LLC. Engines

Funding Sources

- Amana Farms, Inc.
- USDA – Rural Development
- Iowa Office of Energy Independence

For More Information Contact John McGrath at jmcgrath@amanas.net or 319-622-7557

Figure 23. Amana Farms full-scale, mixed plug-flow anaerobic digester quick fact sheet.

Amana Farms Renewable Energy Center & Iowa State University Extension Educational Outreach Program

Anaerobic Digestion Part II: *Light at the End of the Tunnel or a Train?*

Date: Wednesday, October 27, 2010

Location: Iowa Theatre Artists Company
4709 220th Trail
Amana, Iowa 52203

Schedule

9:00 – 9:05 am	Welcome, Introduction John Peterson, Amana Farms
9:05 – 9:15 am	Anaerobic Digestion Principles, Benefits, and Challenges Dr. D. Raj Raman, Iowa State University
9:15 – 9:45 am	Anaerobic Digestion: The Amana Experience John McGrath, Amana Farms
9:45 – 10:15 am	Materials Handling in Anaerobic Digestion Systems Brent Bailey, Vogelsang
10:15 – 10:30 am	*Break*
10:30 – 11:00 am	Digester/Solids Separation Process/Bedding Material Andy Lenkaitis, GEA
11:00 – 11:30 am	Co-digestion Opportunities/Realities: Finding the Right Mix Steven Sell, Iowa State University
11:30 am	Closing Remarks John Peterson, Amana Farms
11:30 – 1:00 pm	Lunch – On Your Own
1:00 – 2:00 pm	Amana Farms Digester Tour

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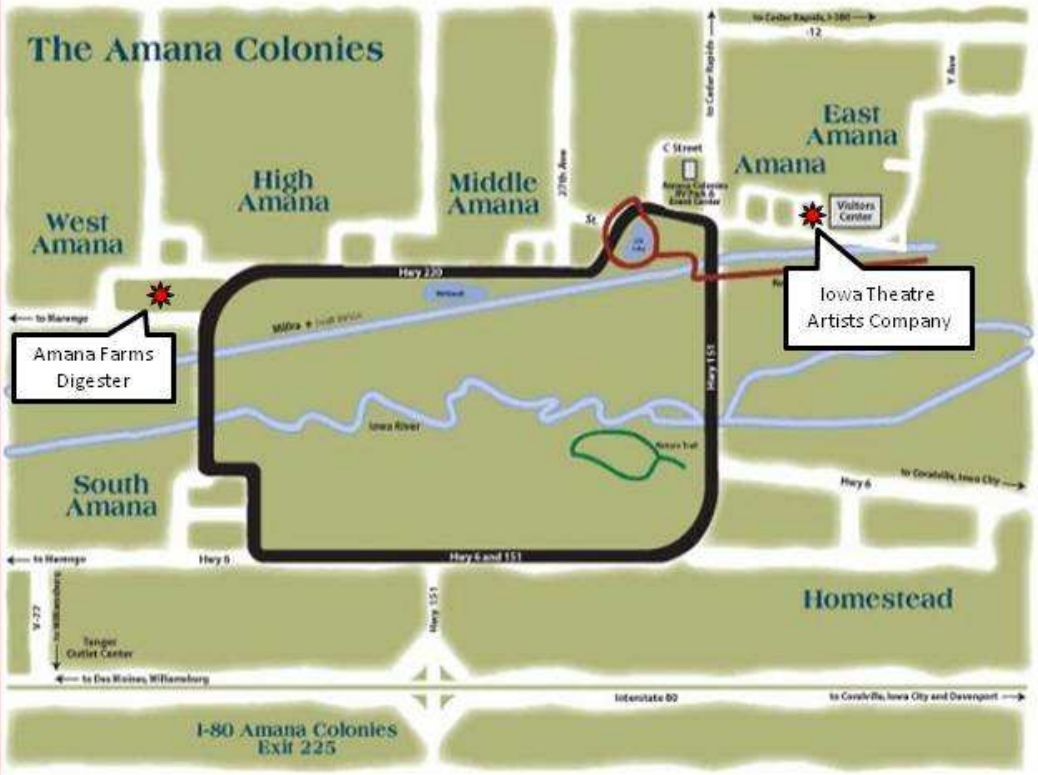
Figure 24. October 27, 2010 Educational Conference Flyer Page One.

Amana Farms Renewable Energy Center & Iowa State University Extension Educational Outreach Program

Anaerobic Digestion Part II:
Light at the End of the Tunnel or a Train?



For Additional Information, contact:
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Steven Sell, stsell@iastate.edu, 515-294-3153

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Figure 25. October 27, 2010 February 16, 2010 Educational Conference Flyer Page Two.

Amana October 2010 Conference Survey

Edit

Default Report | + Add Report

Response Summary

Total Started Survey: 9
Total Completed Survey: 9 (100%)

PAGE:

1. On a scale from 1 - 5, with 1 being very little and 5 being a lot: How much did you learn about the challenges and opportunities of anaerobic digestion?

Create Chart | Download

	Response Percent	Response Count
1	0.0%	0
2	0.0%	0
3	0.0%	0
4	66.7%	6
5	33.3%	3
	answered question	9
	skipped question	0

2. On a scale of 1 – 5, with 1 being very little and 5 being a lot: To what extent did the program address your questions about anaerobic digestion?

Create Chart | Download

	Response Percent	Response Count
1	0.0%	0
2	0.0%	0
3	11.1%	1
4	77.8%	7
5	11.1%	1
	answered question	9
	skipped question	0

Figure 26. Page one of the second educational conference survey.

3. What parts of the program were most valuable to you? Download

		Response Count
Hide Responses		8
1. All the challenges they ran into.	Fri, Nov 12, 2010 5:02 AM	Find...
2. The different waste streams being used in the process interested me most.	Tue, Nov 9, 2010 7:04 AM	Find...
3. The lessons learned from construction of plant, and discovering the mix that generated enough methane.	Tue, Nov 9, 2010 6:09 AM	Find...
4. John's presentation from the Amana perspective	Sat, Nov 6, 2010 4:20 AM	Find...
5. appropriate feedstocks	Fri, Nov 5, 2010 11:50 AM	Find...
6. I enjoyed Andy Lenkaitis' portion of the program. That was a topic I haven't heard discussed much before.	Fri, Nov 5, 2010 8:24 AM	Find...
7. Touring the plant.	Fri, Nov 5, 2010 8:12 AM	Find...
8. Presentation of Amana Farm's experience with their digester.	Fri, Nov 5, 2010 7:48 AM	Find...
	answered question	**8**
	skipped question	**1**

4. What parts of the program were least valuable to you? Download

		Response Count
Hide Responses		8
1. Listening to the equipment reps.	Tue, Nov 9, 2010 7:04 AM	Find...
2. NA	Tue, Nov 9, 2010 6:09 AM	Find...
3. Voglesang's was too technical for this type of conference.	Sat, Nov 6, 2010 4:20 AM	Find...
4. equipment manufactures	Fri, Nov 5, 2010 2:53 PM	Find...
5. chopper pumps	Fri, Nov 5, 2010 11:50 AM	Find...
6. All portions were of value.	Fri, Nov 5, 2010 8:24 AM	Find...
7. Lunch	Fri, Nov 5, 2010 8:12 AM	Find...
8. N/A	Fri, Nov 5, 2010 7:48 AM	Find...
	answered question	**8**
	skipped question	**1**

Figure 27. Page two of the second educational conference survey.

5. What topics would you like to hear more about at the next educational program? Download

		Response Count
Hide Responses		8
1. Specifics about which outsite waste streams are producing the most methane.	Tue, Nov 9, 2010 7:04 AM	Find...
2. The operation and schedule of the ISU simulator digester. It seems the timing can be too long.	Tue, Nov 9, 2010 6:09 AM	Find...
3. The specifics on the generation itself. Cost of equipment, expected payback, life of equipment,	Sat, Nov 6, 2010 4:20 AM	Find...
4. purchases of electricity	Fri, Nov 5, 2010 2:53 PM	Find...
5. the specifics to managing the digestor - what do they monitor on a daily basis, what can they adjust other than the feedstocks introducted	Fri, Nov 5, 2010 11:50 AM	Find...
6. The variances in end products (N,P,K, etc) based on inputs.	Fri, Nov 5, 2010 8:24 AM	Find...
7. Interest in the plant from political leaders. Does US Senator Grassley, a farmer, know this is happening in the Amanas and has he ever seen the plant in operation?	Fri, Nov 5, 2010 8:12 AM	Find...
8. N/A	Fri, Nov 5, 2010 7:48 AM	Find...
	answered question	**8**
	skipped question	**1**

6. Do you have any additional questions or comments? Download

		Response Count
Hide Responses		7
1. Very informational meeting.	Fri, Nov 12, 2010 5:02 AM	Find...
2. No	Tue, Nov 9, 2010 7:04 AM	Find...
3. Overall a good program. Would be nice to get more livestock producers to attend somehow	Sat, Nov 6, 2010 4:20 AM	Find...
4. no	Fri, Nov 5, 2010 11:50 AM	Find...
5. I enjoyed the program very much and hope there will be additional ones in the future.	Fri, Nov 5, 2010 8:24 AM	Find...
6. Would wider advertisement of your scheduled programs be possible? Would efforts toward national recognition be helpful? Job well done. Thanks, – Stan	Fri, Nov 5, 2010 8:12 AM	Find...
7. N/A	Fri, Nov 5, 2010 7:48 AM	Find...
	answered question	**7**
	skipped question	**2**

Figure 28. Page three of the second educational conference survey.