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**Effect of leachate recirculation and use of feedstock specific inoculum on biogas  
production from solid state anaerobic digestion of cattails**

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**ABSTRACT** Solid state anaerobic digestion (SSAD) systems are an attractive option for agricultural waste products such as manure, biomass and other substrates in Western Canada due to the smaller reactor requirements, decreased water usage and utilization of simple batch systems. Collection and utilization of aquatic plants such as cattails (bulrushes) serves several purposes including extraction of nutrients and heavy metals from watersheds, beautification of lakes and rivers and supply of low-cost feedstocks for green energy production. Most studies on the use of aquatic plants for biogas production are based on a liquid-state anaerobic digestion (LSAD) system. In order to achieve maximum capacity from a cost benefit standpoint, SSAD could be a better approach, but basic operating parameters and biogas production values need to be established.

The purpose of this study was to evaluate the biogas production from cattails digested in a solid state anaerobic digester. The study consisted of two bench-scale trials; one to evaluate the effect of weekly leachate recirculation on biogas production, and a second to evaluate the effect of using a feedstock-specific inoculum on biogas production. For each trial, the mixtures consisted of manure only, manure mixed with cattails, and cattails only. In the first trial, each mixture also included a manure digestate as an inoculum (25% by volume). In the second trial, half of the vessels were inoculated with manure digestate and half of the vessels were inoculated with cattail digestate from the first trial.

Substrate- specific inoculum had a positive effect on the enhanced digestion of cattails, increasing biogas production by a factor of six. Weekly leachate recirculation only enhanced gas production in those reactors with cattails only. Gas production and quality from reactors with manure only were similar to previous studies, (73 to 135 L CH<sub>4</sub>/kg VS), while gas production from reactors with cattails only (9 to 104 L CH<sub>4</sub>/kg VS) were lower than from other biomass materials digested in a LSAD system. For SSAD of cattails, it is recommended that a 50% mixture of cattails and manure be used with substrate- specific inoculum. Further testing should be done with respect to leachate recirculation strategies to accurately determine if recirculation is beneficial for this type of substrate.

**Keywords:** anaerobic digestion, solid, inoculum, optimization

## INTRODUCTION

Biogas production via anaerobic digestion (AD) has been regularly used for decades to provide heating and electricity throughout the world through the conversion of organic wastes into energy. Commonly, AD occurs in a liquid-based AD process (LSAD) with a solids content of up to 5%. More recently, solid-state AD systems (SSAD) have been implemented to treat organic and municipal solid wastes (MSW) (DeBaere, 2000; Shafer et al., 2006; Yue et al., 2012). SSAD systems can handle feedstocks with a solids content of 15% to 50%, or an equivalent moisture content of 50% to 85% (Rapport et al., 2008). SSAD systems are an attractive option for agricultural waste products such as manure, biomass and other substrates in Western Canada due to the smaller reactor requirements, lower energy input, decreased water usage and utilization of simple batch systems.

However, issues such as climate in Western Canada pose threats to the implementation of such systems due to high temperature fluctuations and associated vessel sealing issues. Without proper sealing, anaerobic conditions are impossible to maintain and keep methanogenesis proceeding, as well as a loss of methane to the environment (Rapport et al., 2008). The challenge of sealing SSAD systems is compounded by the need to allow for loading and unloading large quantities of solid substrate, usually by a skid steer or front end loader.

Further studies on the effect of climate and other operating hurdles on the sustainability of SSAD are warranted. The Prairie Agricultural Machinery Institute (PAMI) in Humboldt, Saskatchewan, initiated a pilot scale SSAD research program focusing its efforts on the implementation and research associated with biogas production from feedlot manure in Western Canadian climates (Agnew et al., 2013). Others have studied various components of the SSAD system including inoculation strategies (Shafer et al., 2006; Li et al., 2011a; Li et al., 2011b), leachate recirculation (Kusch et al., 2012; El-Mashad et al., 2006; Pratt et al., 2013; Gaudet and Fonstad, 2012) and temperature regimes (Golueke et al., 1957; Parker et al., 2002; Kim et al., 2002) with mixed results. For implementation of this technology in Western Canada, a variety of parameters such as initial inoculum concentration and type, leachate recirculation strategies and various substrates' biogas potential should be analyzed and best management practices developed.

Renewable biomass materials such as aquatic plants used for wastewater cleanup or invasive species in lakes and rivers could be harvested and implemented in the AD process. Collection and utilization of aquatic plants such as cattails (bulrushes) serves several purposes including extraction of nutrients and heavy metals from watersheds, beautification of lakes and rivers and supply of low-cost feedstocks for green energy production. Most studies on the implementation of aquatic plants as feedstocks for AD are based on a LSAD system. In order to achieve maximum capacity from a cost benefit standpoint, high solids, dry AD could be a better approach. The basic technology for this type of AD exists, but many improvements are necessary in terms of optimization, substrate mixes and end product uses (Karthikeyan et al., 2012).

There is little literature on the study of AD using cattails as a substrate; the studies that do exist are aimed at optimizing the acidogenesis stage by rumen cultures to produce volatile fatty acids (VFAs) (Yue et al., 2009; Yue et al., 2013; Hu et al., 2012; Hu et al., 2006). Others have studied liquid state AD of cattails specifically combined with cockroach gut microorganisms to produce methane via AD at 30°C (Mshandete, 2009). Mshandete (2009) observed methane production between 288 – 447 mL/g VS dependent on the portion of the plant digested, with leaves providing higher production than the whole plant. Similarly, others have studied AD of other aquatic plants as substrates with mixed results. In a study comparing digestion of bulrush plants with two inoculum types (rumen cultures and digester sludge), a higher methane yield was

observed in the reactors inoculated with digester sludge than those with rumen cultures (Yue et al., 2012). Due to the nature of cattails, it could be beneficial to compare across other plants of similar lignocellulosic biomass. Table 1 demonstrates some results found in other studies of liquid state AD of similar plants.

Table 1. Product and methane yield from liquid state anaerobic digestion of lignocellulosic feedstocks.

Substrate	Product Yield mg COD/g VS	Methane Yield L/kg VS	Reference
Bulrush	362-464		Yue et al., 2012
Corn stover	448-672	410	Hu and Yu, 2005; Liew et al., 2012
Maize Bran	530-600		Kivaisi, 1995
Paper tube residuals	603		Teghammar et al., 2010.
Bagasse	544-610	300	Kivaisi, 1995; Quintero et al., 2012; Escalante et al., 2014
Cattails		288-447	Mshandete, 2009
Wheat Straw		66.9	Liew et al., 2012
Leaves and yard waste		40.8-55.4	Liew et al., 2012
Corn Silage		296	Labatut et al., 2011
Switchgrass		122	Labatut et al., 2011
Water Chestnut		359	Labatut et al., 2011
Water Celery		384	Labatut et al., 2011
Frogbit		451	Labatut et al., 2011
Sugarcane		266-314	Chynoweth et al., 1993

The goal of this study was to assess biogas potential from dried and chopped cattails (*Typha spp.*) under mesophilic (38-40°C) conditions using a SSAD system. As there is limited information available on optimal operating conditions for SSAD of cattail feedstock, this project focused on establishing baseline information on biogas yield, the effect of weekly leachate recirculation on biogas production potential and effect of using a feedstock specific inoculum as a seed material on biogas production potential. Since previous SSAD work showed that co-digestion of biomass with manure generated more biogas than digestion of biomass alone, the effect of mixing cattail with solid feedlot manure on biogas production was also evaluated.

## MATERIALS AND METHODS

The project consisted of two bench-scale SSAD trials conducted at the University of Saskatchewan in Saskatoon, Saskatchewan.

### ***Incubator Set-up***

The bench scale SSAD system constructed at PAMI and the University of Saskatchewan was used to assess biogas production and quality for this study (Pratt et al., 2013; Gaudet and Fonstad, 2012). A total of 18 vessels, each with a capacity of 3-4 kg solid substrate were held in two incubators (Figure 1) equipped with heating units capable of maintaining 38-40°C. Each

vessel was equipped with a thermocouple, gas line and leachate recirculation line. Gas production was measured with wet tip gas meters (*Wet Tip*) calibrated to tip for every 80 mL of gas produced. A data logging system (*DataTaker D80*) was used to record temperatures via thermocouple (in-vessel, incubator, and room), barometric pressure, and gas tip events. Upon loading, initial substrate weights and headspace volume were recorded and a sub-sample of each mix sent to ALS Laboratories (Saskatoon, SK) for analysis of moisture content, electrical conductivity, total carbon by combustion, loss on ignition (volatile solids content), total nitrogen (N), ammonia-N, nitrate-N, pH, total phosphorous (P), potassium (K) and sulphur (S) by standard methods. After loading, approximately one liter of water was added to each vessel to reach saturation. At the end of the trial, leachate was evacuated, vessels were weighed and a subsample of each mix was again sent to ALS Laboratories for the same analysis as the initial samples.



Figure 1. Incubator set-up with nine vessels. Two incubators are used for a total of 18 vessels.

### **Gas Analysis**

Gas samples were collected from each vessel on day three and weekly for the remainder of each trial. A total of 30 mL was purged via syringe from the sample line before a 15 mL gas sample was drawn and placed into a vacutainer for gas chromatography (GC) analysis for percent carbon dioxide (CO<sub>2</sub>) and percent methane (CH<sub>4</sub>). Gas samples were analyzed for methane using a Varian CP-3800 gas chromatograph, while carbon dioxide was measured using a Varian Micro-GC CP-2003 with a sample time of 30 seconds and a 180 second run time. Carbon dioxide was detected using a thermal conductivity detector (TCD) coupled with a Poraplot U column 10 m in length with a 0.32 mm inner diameter. Column temperature for the TCD was 45°C with an injector temperature of 55°C with an injection time of 25 ms under 100 kPa. The detection limit for CO<sub>2</sub> measurement with this device is 80 ppm. The detector used for methane analysis was a flame ionization detector (FID) which uses a Poraplot Q8 column that is 3.65 m in length with a 3.175 mm diameter and 2 mm film thickness. Column temperature for the FID was 45°C with an injector temperature of 55°C with an injection time of 20 ms under 140 kPa. The detection limit for methane on this instrument was 360 ppb. The carrier gas used in all detectors was helium for both CO<sub>2</sub> and CH<sub>4</sub> measurement.

## ***Substrate Analysis***

Subsamples of each substrate mix pre- and post-AD were sent to ALS Laboratories (Saskatoon, SK) for analysis. Moisture content was determined using American Society for Testing and Materials (ASTM) D2216-80 (ASTM, 2012) and total carbon, phosphorus, potassium and sulphur by Soil Science Society of America Standards (SSSA, 1996). Ammonia-N and nitrate-N were determined using standards from Canadian Society of Soil Science standards (Carter, 1993) while total N using RMMA A3769 3.3 (Wolf et al., 2005). Test Methods for the Examination of Composting and Compost standards (Leege and Thompson, 2001) were used to determine EC (4. 10-A), loss on ignition to determine volatile solids (VS) (05.07-A) and pH (4.11-A).

## ***Feedstock and Inoculum***

Cattails were harvested in October, 2013 from Pelly's Lake near Holland, Manitoba, then air-dried and chopped to 2 to 5 cm lengths. Beef feedlot manure was collected from the Western Beef Development Center feedlot in April, 2014 from pens that used woodchips for bedding. Inoculum for trial 1 was digestate from a previous AD trial utilizing solid feedlot manure, and inoculum for trial 2 was digestate from the first cattails AD trial (cattail specific inoculum) and digestate from a pilot-scale SSAD trial (feedlot manure specific inoculum).

The first trial consisted of three treatment mixtures (by volume):

Mix 1. Manure (75%) with manure digestate (25%) (control)

Mix 2. Cattails (37.5%) with manure (37.5%) and manure digestate (25%), and

Mix 3. Cattails (75%) with manure digestate (25%).

Each mixture was loaded into six vessels. Three vessels of each mixture received weekly leachate recirculation to determine the effect of recirculation on biogas production. This trial showed initial failures and a severe lag in biogas production, so the trial run-time was extended to 117 days since others have shown that if you let the trial run past 60 days, the reactors may start to recover (Town et al., 2014). **Figure 2** shows before and after for the cattail/manure/digestate mixture.



Figure 2. Trial 1, mix 2, before (L) and after (R) AD.

Based on the results from the first trial, the second trial was used to determine if substrate specific inoculum improved biogas production from non-manure feedstocks. For this trial,

manure digestate inoculum was compared to cattail digestate inoculum. The reactor mixes were prepared in a similar manner as the first cattails trial, with each mix receiving either cattail specific inoculum or manure specific inoculum (25% by volume). For this trial, the incubation period was 42 days. This second trial did not have any leachate recirculation. The pre- and post-AD feedstock appeared similar to the feedstock in Trial 1 (Figure 3).



Figure 3. Trial 2, mix 2 before (L) and after (R) AD.

## **RESULTS AND DISCUSSION**

The results from both bench-scale trials are presented here.

### ***Trial 1***

Biogas production for the first few days of the trial showed a sharp peak and then significant tapering and lagging of biogas production. This lag time could have been caused by bad inoculum or acid stall in the process. Approximately 65 days went by before the reactors started to recover and produce biogas (Figure 4). The highest producer of biogas was mix 1 (manure), followed closely by mix 2 (manure/cattail) while the cattail vessels produced significantly less biogas than the other vessels.

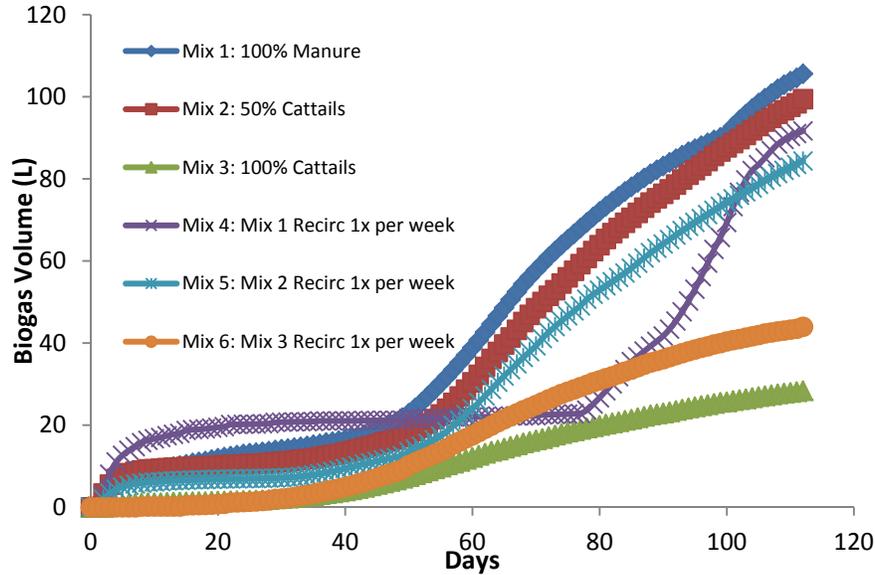


Figure 4. Cumulative biogas production from Trial 1.

Leachate recirculation increased biogas production from the cattail vessels but hindered biogas production from the manure and manure/cattail vessels. Previous work with SSAD has also shown that leachate recirculation produces mixed results in terms of biogas production (Pratt et al., 2013; Pratt et al., 2014) although recirculation generally improves biogas production from non-manure feedstocks. Table 2 summarizes the key parameters observed with respect to biogas production, quality and rates on a L/kg volatile solids (VS) basis. Biogas production rates per kg VS were significantly higher for mix 1 than mix 2 and 3. Due to the lignin and cellulose composition of the cattails, this was to be expected. Quality of biogas is of concern for this trial, as the CO<sub>2</sub> and CH<sub>4</sub> made up only 60% of the total gas measured in the first part of the trial. No other gases were measured, but the remaining 40% could have been hydrogen or nitrogen gas. Further into the trial, combined concentrations reached closer to 80% of the total, similar to other studies. For all cases, the cattail/manure mixture produced higher quality biogas (higher CH<sub>4</sub> concentration) than the cattail alone. Based on the results of this trial, SSAD of cattails alone does not produce enough biogas in a quality that is acceptable for conversion into bioenergy.

Table 2. Gas production results from Trial 1.

Summary	Mix 1: Manure	Mix 2: Cattails/manure	Mix 3: Cattails	Mix 1R	Mix 2R	Mix 3R
Total Gas Production (L)	100.7	100.4	29.0	92.5	85.3	63.5
Average Methane Composition (%)	33.8	40.0	30.0	39.5	35.8	50.1
Biogas Production Rate (L/kg VS)	214.6	82.8	29.0	198.0	71.5	73.6
Methane Rate (L/kg VS)	72.6	33.1	8.7	78.3	25.6	36.9

## Trial 2

The second trial assessed whether or not substrate-specific inoculum improved biogas production from a non-manure feedstock. Manure inoculum was acquired from the PAMI pilot scale SSAD and compared to the cattails digestate from Trial 1. Leachate recirculation was not done for this trial, as the trial was a pure assessment of whether “substrate specific” inoculum would produce the most biogas. The trial was successful and ran for a total of 45 days. All mixes with cattails and “cattails inoculum” produced significantly more biogas than their “manure inoculum” counterpart (Figure 5). For example, the 50% cattails inoculated with cattails digestate produced 125 L of biogas, while its counterpart, only 111; for the 100% cattails mix, 103 L vs 26 L. The 100% manure vessels produced more biogas with the manure inoculum (162 L) than the cattail inoculum (137 L). Basically, this is suggesting that in order to optimize biogas production from specific substrates, a “substrate specific” inoculum is important.

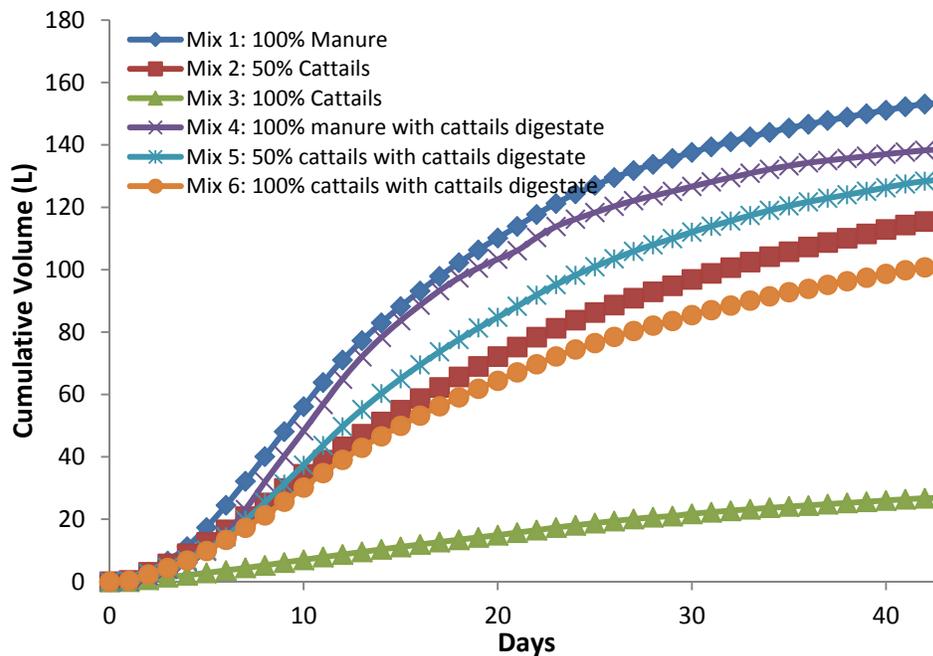


Figure 5. Cumulative biogas production for each mixture in Trial 2.

The biogas quality from Trial 2 was better than the quality from Trial 1. Trial 2 produced a maximum ratio of nearly 60/40 ( $\text{CH}_4/\text{CO}_2$ ) by day 14 and tapered off slightly for the remainder of the trial. The best quality of gas was again produced by manure (mix 1) and cattail/manure, with cattails only producing the lowest quality. Interestingly, when comparing Trial 1 characteristics of gas production to Trial 2 (Table 3), the amount of gas produced and rate per kg VS is not very different except in the case of 100% cattails inoculated with cattails digestate. In fact, the biogas production from cattails inoculated with manure digestate was the same for both trials, but the biogas production from cattails inoculated with cattail digestate increased by a factor of six.

Table 3. Trial 2 gas production characteristics.

<b>Trial Summary</b>	<b>Mix 1: Manure with manure digestate</b>	<b>Mix 2: Cattails/m anure with manure digestate</b>	<b>Mix 3: Cattails with manure digestate</b>	<b>Mix 4: 100% Manure with cattails digestate</b>	<b>Mix 5: 50% Cattails/ma nure with cattails digestate</b>	<b>Mix 6: 100% Cattails with cattails digestate</b>
Total Gas Production (L)	162	111	27	137	125	104
Average Methane Composition (%)	41	37	27	42	40	50
Biogas Production Rate (L/kg VS)	330	145	28	268	146	97
Methane Rate (L/kg VS)	135	54	8	112	58	49

The effect of AD on composition of substrate (moisture content, volatile solids, nutrient content, etc.) is shown in the Appendix.

### ***Comparison to Other Studies***

The results from this trial indicated a range of methane production of 33 to 125 L CH<sub>4</sub>/kg VS for cattail mixed with manure and a range of 9 to 104 L CH<sub>4</sub>/kg VS for cattail only. Comparing these values with those found in literature (Table 1), it is apparent that and the yields from this trial are lower that what others have found for cattails in a LSAD system (Mshandete, 2009). Results from SSAD of other feedstocks like feedlot manure show a similar trend; biogas production on a per kg VS solids basis is lower for SSAD than LSAD, likely due to the enhanced mixing and smaller particle sizes in a LSAD system. However, the reactor volume (and capital cost) is much lower for a SSAD system. Therefore, the biogas production on a per L reactor basis should also be calculated for future comparisons.

### **CONCLUSION**

A comparison of leachate recirculation strategies and substrate specific inoculum was performed on dried and chopped cattails in various mixtures of manure and digestate. Substrate specific inoculum had a positive effect on the enhanced digestion of cattails, but did not aid in the digestion of 100% manure. Weekly leachate recirculation enhanced gas production in those reactors with 100% cattails, but did not appear to improve gas production from other mixtures. Gas production and quality from reactors with only manure were similar to previous studies (73 to 135 L CH<sub>4</sub>/kg VS) while gas production from reactors with only cattails (9 to 104 L CH<sub>4</sub>/kg VS) were lower than from other biomass materials digested in a LSAD system. For SSAD of cattails, it is recommended that a 50% mixture of cattails and manure be used with substrate specific inoculum. Further testing should be done with respect to leachate recirculation strategies to truly decide which is more appropriate for this type of substrate.

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## APPENDIX

Summary of feedstock composition pre- and post-digestion for Trial 2.

		Pre-digestion						Post-digestion					
		Mix 1	Mix 2	Mix 3	Mix 4	Mix 5	Mix 6	Mix 1	Mix 2	Mix 3	Mix 4	Mix 5	Mix 6
Moisture	%	85	51	34	84	50	33	79	80	71	77	82	82
Ammonia as N	kg/tonne	2.91	1.46	0.73	2.76	1.31	0.58	0.75	0.48	0.39	0.86	0.61	0.53
Available Phosphate-P	mg/kg	5885	2799	1255	5627	2541	998	2650	3370	2170	2660	966	1030
Loss on Ignition at 550°C	%	79	86	90	80	87	91	46	77	81	58	81	81
Total Carbon by Combustion	kg/tonne	75	236	316	77	238	318	75	78	141	66	81	94
Total Nitrogen	kg/tonne	6.14	8.42	9.56	6.16	8.44	9.58	5.08	5.03	4.70	4.73	4.47	4.95
pH (1:2 soil:water)	pH	8.29	8.33	8.35	8.35	8.39	8.41	9.08	9.56	9.54	9.48	9.41	9.36
Phosphorus (P)	kg/tonne	1.42	1.03	0.83	1.43	1.03	0.83	1.02	1.04	0.75	1.41	0.49	0.79
Potassium (K)	kg/tonne	3.60	3.23	3.04	3.69	3.31	3.12	2.75	2.76	2.34	3.38	1.75	1.66
Sulfur (S)	kg/tonne	0.94	1.57	1.88	0.96	1.59	1.90	0.49	0.72	0.71	0.62	0.50	0.53

Mix 1: manure with manure inoculum

Mix 2: cattail/manure with manure inoculum

Mix 3: cattail with manure inoculum

Mix 4: manure with cattail inoculum

Mix 5: cattail/manure with cattail inoculum

Mix 6: cattail with cattail inoculum

