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# Effect of limited air exposure and comparative performance between thermophilic and mesophilic solid-state anaerobic digestion of switchgrass

Johnathon P. Sheets, Xumeng Ge, Yebo Li *

*Department of Food, Agricultural and Biological Engineering, The Ohio State University/Ohio Agricultural Research and Development Center, 1680 Madison Ave., Wooster, OH 44691-4096, USA*

## HIGHLIGHTS

- Mesophilic and thermophilic solid-state anaerobic digestion (SS-AD) of switchgrass.
- Limited air exposure to SS-AD digester headspace did not affect SS-AD performance.
- Increasing TS from 20% to 30% negatively affected volumetric methane productivity.
- Thermophilic SS-AD had about 30% greater methane yield than mesophilic SS-AD.
- Optimal digestion periods for SS-AD of switchgrass are 25–35 days.

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## ABSTRACT

Switchgrass is an attractive feedstock for biogas production via anaerobic digestion (AD). Many studies have used switchgrass for liquid anaerobic digestion (L-AD), but few have used switchgrass for solid-state anaerobic digestion (SS-AD). Limited air exposure to the reactor headspace has been adopted in commercial scale anaerobic digesters for different applications. However, little research has examined the effect of limited air exposure on biogas production during SS-AD. In this study, the effects of air exposure and total solids (TS) content on SS-AD performance were evaluated under mesophilic (36 ± 1 °C) and thermophilic (55 ± 0.3 °C) conditions. Limited air exposure did not significantly influence the methane yield during SS-AD. Thermophilic SS-AD had greater methane yields (102–145 L $CH_4$ $kg^{-1}$ $VS_{added}$) than mesophilic SS-AD (88–113 L $CH_4$ $kg^{-1}$ $VS_{added}$). Both mesophilic SS-AD (73–136 GJ) and thermophilic SS-AD (2–95 GJ) produced positive net energy based on a theoretical 'garage-type' SS-AD digester operating in a temperate climate.

## 1. Introduction

The United States Department of Energy (U.S. DOE) considers switchgrass (*Panicum virgatum* L.) as a model lignocellulosic energy crop, due to its high productivity, efficient water and nutrient use, and adaptability to marginal lands (Keshwani and Cheng, 2009). Anaerobic digestion (AD) is a robust process that is able to convert complex organic material such as energy crops into biogas composed of 60–70% methane (Barbanti et al., 2014). Biogas is a flexible renewable fuel that can be upgraded into transportation fuels or used directly to generate electricity and heat (Li et al., 2011). In fact, biogas from AD has recently been included in the United States Renewable Fuels Standard (US EPA, 2014), and switchgrass has been tested as a feedstock in liquid-AD (L-AD) systems (Frigon et al., 2012; Massé et al., 2010), which use low total solids (TS) contents of less than 15% (Xu et al., 2014). However, floating and stratification of fibrous materials has made L-AD of lignocellulosic biomass difficult to scale up (Frigon and Guiot, 2010). In contrast, solid-state anaerobic digestion (SS-AD) systems operate at TS contents greater than 15%. Compared to L-AD, SS-AD has higher volumetric productivities (Li et al., 2011) and generates a digestate that is easier to transport due to low moisture (Xu et al., 2014). These advantages make SS-AD an intriguing option for bioenergy production from lignocellulosic feedstocks, such as switchgrass. However, research on SS-AD of switchgrass is limited (Ahn et al., 2010; Brown et al., 2012; El-Mashad, 2013). Prior to the scale up of this promising approach, it is essential to evaluate the performance of SS-AD of

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* Corresponding author. Tel.: +1 330 263 3855.
*E-mail address:* li.851@osu.edu (Y. Li).

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switchgrass under different environmental conditions, such as limited air exposure and operating temperature.

A common practice in L-AD systems is to supply oxygen ($O_2$) or air to the digester headspace during operation. Limited aeration may lead to increased rates of hydrolysis and fermentation, likely because many fermentative AD microbes are facultative anaerobes (Botheju and Bakke, 2011). In fact, several studies showed that limited air addition did not inhibit the strictly anaerobic methanogens during L-AD, and actually enhanced methane yield (Díaz et al., 2010; Lim and Wang, 2013). In addition, limited aeration is also utilized to remove hydrogen sulfide ($H_2S$) in the biogas prior to upgrading (Díaz et al., 2010). Therefore, limited air exposure could be a helpful process to improve both biogas production and quality in SS-AD. However, there is little research available on the effect of air exposure on biogas production during SS-AD (Charles et al., 2009).

SS-AD can operate under either mesophilic (37 °C) or thermophilic (55 °C) conditions. Mesophilic AD is better established, and often more stable and reliable than thermophilic AD (Labatut et al., 2014). However, thermophilic conditions have been shown to accelerate the conversion of organic material into biogas during SS-AD (Li et al., 2011). Thermophilic temperatures may result in inhibitory levels of volatile fatty acids (VFAs) due to enhanced activity of fermentative microbes (Ahn et al., 2010; Labatut et al., 2014; Shi et al., 2013), which can be addressed by controlling the feedstock to inoculum (F/I) ratio and carbon to nitrogen (C/N) ratio, and providing proper nutrients and pH buffering (Lin et al., 2014; Shi et al., 2014). One major concern about thermophilic SS-AD is the high energy input required to maintain the thermophilic temperature, which may vary significantly for different seasons and can offset the high biogas production rates (Li et al., 2011). To the best knowledge of the authors, there have been no reports that evaluated the performance between mesophilic and thermophilic SS-AD of switchgrass based on energy inputs and outputs while operating at elevated TS contents (>20%).

To address these research gaps, the objectives of this study were to: (1) evaluate the effect of limited air exposure on biogas production during SS-AD; (2) compare performance of mesophilic and thermophilic SS-AD of switchgrass at elevated TS contents; and (3) determine the net energy production during mesophilic and thermophilic SS-AD of switchgrass during long-term operation.

## 2. Methods

### 2.1. Feedstock and inoculum

Switchgrass (*Panicum virgatum L.*, cultivar: Cave-in-Rock) was collected in October 2009 from a farm located at the Ohio Agricultural Research and Development Center (OARDC) in Jackson, Ohio. All experiments were done at the OARDC in Wooster, Ohio. Upon receipt, switchgrass was oven dried at 40 °C in a convection oven (Precision Thelco Model 18, Waltham, MA, USA) to less than 10% moisture, ground with a hammer mill (Mackisik, Parker Ford, PA, USA) to pass through a 5 mm screen, and stored in air-tight containers prior to use. Switchgrass was composed of 91.3 ± 0.2% TS, 96.9 ± 0.0% volatile solids (VS, based on TS), 3.1 ± 0.0% ash (based on TS), 43.7 ± 0.7% total carbon (TC), and 0.6 ± 0.1% total nitrogen (TN). The extractives, cellulose, hemicellulose, and lignin contents were 12.1 ± 1.2%, 31.0 ± 1.0%, 19.5 ± 0.6%, and 19.3 ± 0.4%, respectively (all based on TS of sample).

Raw effluent (TS = 7.7%) was collected from a commercial scale, mesophilic liquid anaerobic digester (KB Compost Services, Akron, OH, USA) which uses municipal sewage sludge as a feedstock. Raw effluent was dewatered by centrifugation to increase the TS from 7.7% to 17.8%. Raw and centrifuged effluents were stored in air-tight buckets at 4 °C prior to use. Proportions of raw and centrifuged effluent were activated anaerobically for one week at 36 ± 1 and 55 ± 0.3 °C prior to inoculation in mesophilic and thermophilic SS-AD reactors, respectively.

### 2.2. Solid-state anaerobic digestion

The effects of TS, temperature, and air exposure on the performance of SS-AD were evaluated using a three factor-two level ($2^3$) experimental design. Two levels of TS (20%, 30%), temperature (36 ± 1, 55 ± 0.3 °C), and air exposure (no exposure, limited exposure) were used for the SS-AD experiments. Three replicate reactors were designed for each treatment combination (24 total reactors). The raw L-AD effluent, centrifuged L-AD effluent, and switchgrass were thoroughly mixed to the desired TS at a feedstock to inoculum (F/I) ratio of 3 (based on VS). For reactors at 20% TS, switchgrass, raw effluent, and centrifuged effluent represented 64%, 31%, and 8% of the reactor content (dry basis), respectively. For reactors at 30% TS, switchgrass, raw effluent, and centrifuged effluent represented 64%, 8%, and 28% of the reactor content (dry basis), respectively. Pre-mixing was conducted under aerobic conditions. SS-AD was carried out in 1-L glass reactors, and each was loaded with 400 g (30% TS) to 550 g (20% TS) of mixed materials to reach an initial working volume of ~850 mL. After loading, half of the reactors were purged with pure nitrogen ($N_2$) gas for 1 min to induce oxygen-depleted conditions (initial $O_2$ content < 0.3%), then sealed with a rubber stopper. The remaining reactors were simply sealed with a rubber stopper after mixed materials were loaded. All reactors were then placed in either a 36 ± 1 °C or 55 ± 0.3 °C incubator for mesophilic and thermophilic SS-AD, respectively. The full digestion period was 70 days. On days 10, 20 and 30, 100 mL of headspace gas was displaced by ambient air using a plastic syringe. This was only conducted for unpurged reactors. Biogas was collected in 5-L Tedlar gas bags (CEL Scientific, Santa Fe Springs, CA) attached to a single outlet on each reactor. For the first 35 days, biogas composition and volume were measured every 2–4 days. From day 35 to day 55, biogas composition and volume were measured once per week. As controls, reactors loaded only with centrifuged L-AD effluent were run in parallel. One of the $N_2$ purged reactors under thermophilic conditions (TS = 20%) had seal failure on day 15, and was not included in data analysis.

### 2.3. Analytical methods

Compositional analysis of switchgrass, raw L-AD effluent, centrifuged L-AD effluent, mixed materials before SS-AD, and digestate after SS-AD was conducted. The TS, VS, pH, and alkalinity were measured according to Standard Methods for the Examination of Water and Wastewater (APHA, 2005). Samples for volatile fatty acid (VFA) measurement were prepared using an adapted version of the methods described by Shi et al. (2013), which consisted of suspending 5 g of L-AD effluent or digestate in 5 mL of deionized water, thoroughly mixing it, and separating the solids by centrifugation (10,000 rpm for 5 min). The supernatant was acidified to a pH of 2–3 by addition of hydrochloric acid, and then filtered via a syringe filter (0.2 μm). VFAs (acetic, propionic, isobutyric, butyric, isovaleric, valeric acids) were measured using a gas chromatograph (GC) (Shimadzu, 2010 PLUS, Columbia, MD, USA) equipped with a 30 m × 0.32 mm × 0.5 μm Stabilwax polar phase column and flame ionization detector. Total carbon (TC) and total nitrogen (TN) were measured with an elemental analyzer (Vario Max CNS, Elementar Americas, Mt. Laurel, NJ, USA) in order to calculate the C/N ratio. Total ammonia nitrogen (TAN), composed of free ammonia ($NH_3$) and ammonium ($NH_4^+$), was measured by a modified distillation and titration method (ISO 5664, 1984) that used 4% boric acid and a Kjeldahl Distillation Unit B-324 (Buchi, Labortechnik, AG, Switzerland). Extractives of raw materials and SS-AD digestate were measured according to the NREL Analytical Procedure