



Anaerobic digestion of giant reed for methane production



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HIGHLIGHTS

- Anaerobic digestion (AD) of giant reed was at various total solids (TS) contents.
- Increasing TS from 8% to 38% negatively affected methane yield.
- Highest volumetric methane production was obtained at 20–23% TS.
- Methane yield from solid state AD was 16% lower than that from liquid AD at $F/E = 2.0$.
- Cellulose showed the highest contribution to methane production.

ARTICLE INFO

Article history:

Received 1 July 2014
Received in revised form 7 August 2014
Accepted 9 August 2014
Available online 18 August 2014

Keywords:

Solid-state anaerobic digestion
Giant reed
Methane
Cellulose
Total solids

ABSTRACT

As a fast growing plant, giant reed has good potential to be used as a feedstock for methane production via anaerobic digestion (AD). The effect of total solids (TS) content, an AD operating parameter, was studied. Results showed that increasing TS from 8% to 38% decreased methane yield, due to the inhibition of volatile fatty acids (VFAs) and total ammonia nitrogen (TAN); while the maximum volumetric methane production was obtained at 20–23% TS. Comparison of solid-state AD (SS-AD) at 20% TS and liquid AD (L-AD) at 8% TS was conducted at feedstock to effluent (F/E) ratios of 2.0, 3.5, and 5.0. The best performance was achieved at an F/E of 2.0, with methane yields of 129.7 and 150.8 L-CH₄/kg-VS for SS-AD and L-AD, respectively. Overall organic components were degraded by 17.7–28.5% and 24.0–26.6% in SS-AD and L-AD, respectively; among which cellulose showed the highest degradation rate and the highest contribution to methane production.

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1. Introduction

Lignocellulosic biomass is considered to be a promising feedstock for bioenergy production due to its abundance, renewability, and the fact that it does not compete with food or feed production. Therefore, in recent years, efforts have been made to identify new sources of lignocellulose for bioenergy production (Zheng et al., 2014), among which, giant reed (*Arundo donax* L.) is gaining increasing interest. Giant reed is a perennial weed grass plant, and is usually cultivated in subtropical and warm temperate regions. The attractive features of giant reed for bioenergy production are its high growth rate, suitability for harvesting more than once per year, and tolerance to dry environments (Pilu et al., 2013). A previous study reported that giant reed could have a growth rate of about 5 cm per day in favorable environments (Pilu et al., 2013). In Central Italy, a 10-year study showed a higher

dry mass production rate of giant reed (37.7 ton per year per hectare) than of miscanthus (28.7 ton per year per hectare) (Angelini et al., 2009). Due to its high growth rate, giant reed can be harvested twice per year (harvest–regrow–harvest), producing 20% more biomass than that obtained from a single harvest (Ragagnoli et al., 2014a). Giant reed is considered to be a drought-tolerant species and can adapt to marginal or sub-marginal lands, thus not competing with food crops for land (Angelini et al., 2009). Currently, giant reed is widely planted in East Asia, Mediterranean regions, and both East and West coasts of the U.S., and is primarily used as a source of fiber for printing paper (Fiore et al., 2014), an alternative to wood to make chipboard panels (Flores et al., 2011), or a raw material for manufacturing activated carbon (Sun et al., 2012). Giant reed has also been examined as a feedstock for bioenergy production. A few studies have investigated fermenting giant reed for ethanol production (Scordia et al., 2012, 2013). Anaerobic digestion (AD) has been employed to produce biogas from giant reed. Ragagnoli et al. (2014b) showed the highest biochemical methane potential of giant reed to be 392 L per kg

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volatile solids (VS), which is equivalent to a methane yield of 11,585–12,981 m³ ha⁻¹ based on the biomass yield per hectare. [Girolamo et al. \(2013\)](#) pretreated giant reed using hydrothermal methods with sulfuric acid (H₂SO₄) as the catalyst, and increased methane yield by 4–23%. According to these studies, AD is an effective method for extracting bioenergy from giant reed that is reliable and has low greenhouse gas emissions.

While most previous studies have employed liquid AD (L-AD) to digest lignocellulosic biomass, the high solids content in lignocellulosic biomass has spurred researchers to consider solid-state AD (SS-AD) to digest giant reed. Compared to L-AD, which is usually operated with less than 15% total solids (TS) content, SS-AD can handle high solids containing feedstocks and operates with TS higher than 15%. Consequently, compared to L-AD, SS-AD usually has a higher organic loading rate, smaller reactor volume, lower energy demand for heating, higher volumetric methane productivity, and less wastewater generation ([Li et al., 2011](#)). To date, no SS-AD of giant reed has been carried out and research is needed to identify the optimal TS range for SS-AD of giant reed. It is also important to know the contribution of each organic component, e.g. cellulose, hemicellulose, and protein, to methane production during SS-AD. Therefore, the objectives of this study were to examine the effects of TS on methane production from AD of giant reed; compare the performance of SS-AD and L-AD in digesting giant reed; and estimate the contribution to methane production from each organic component in giant reed.

2. Methods

2.1. Feedstock and inoculum

Giant reed was planted in April 2013 at a research farm near Columbus, OH, and was harvested in October 2013. Upon receipt, giant reed was ground through a 12 mm sieve with a grinder (Mighty Mac, Mackissic Inc., Parker Ford, PA, USA). To increase the total solids content, fresh giant reed was air dried in a 40 °C oven for 2 days. Both fresh and dried samples were used as feedstocks for the AD tests. To inoculate the feedstock, effluent taken from a mesophilic liquid anaerobic digester (operated by *quasar energy group* in Zanesville, OH, USA) was centrifuged and then well mixed with the feedstock. Centrifuge permeate was used to adjust the overall TS content. Characteristics of the feedstocks and effluent are presented in [Table 1](#).

Table 1
Properties of initial feedstocks and inoculum.

Parameters	Fresh feedstock	Dried feedstock	Centrifuged inoculum ^a
TS, %	37.64 ± 0.54	96.63 ± 0.20	22.19 ± 1.41
VS, %	34.43 ± 0.59	88.43 ± 0.27	12.43 ± 0.68
TC, %	18.30 ± 1.93	42.75 ± 0.67	6.27 ± 0.12
TN, %	0.54 ± 0.07	1.24 ± 0.03	0.66 ± 0.02
C/N ratio	34.31 ± 1.04	34.47 ± 0.42	9.47 ± 0.17
pH	7.68 ± 0.07	7.06 ± 0.03	8.00 ± 0.01
Alkalinity, g-CaCO ₃ /kg	2.39 ± 0.57	1.99 ± 0.08	10.76 ± 0.21
VFAs, g/kg	0.48 ± 0.02	1.24 ± 0.04	0.93 ± 0.06
TAN, g-N/kg	1.29 ± 0.11	1.67 ± 0.12	5.52 ± 0.15
Extractives ^b , %	19.27 ± 0.32	19.27 ± 0.32	15.02 ± 0.27
Cellulose ^b , %	20.62 ± 3.59	20.62 ± 3.59	1.45 ± 0.28
Hemicellulose ^b , %	6.88 ± 1.57	6.88 ± 1.57	0.86 ± 0.02
Lignin ^b , %	33.85 ± 1.83	33.85 ± 1.83	N/A
Crude protein ^b , %	8.39 ± 0.27	6.67 ± 0.14	8.70 ± 0.24

TS = total solids, VS = volatile solids, TC = total carbon, TN = total nitrogen, C/N = ratio of carbon over nitrogen, VFAs = volatile fatty acids, TAN = total ammonia nitrogen.

Average ± S.E., n = 2.

^a Centrifuged at 10 k rpm for 10 min.

^b Based on TS, others are based on total weight.

2.2. Anaerobic digestion

To examine the effect of TS on AD performance, dried feedstock was mixed with inoculum at a feedstock to effluent (F/E, based on VS) ratio of 2.0 with TS contents of 38%, 33%, 28%, and 23%; while fresh feedstock was inoculated at the same F/E ratio with TS contents of 28%, 23%, 18%, 13%, and 8%. To compare SS-AD and L-AD performance, fresh giant reed was inoculated to achieve F/E ratios of 2.0, 3.5, and 5.0 and the TS was controlled at 20% and 8% for SS-AD and L-AD, respectively. L-AD reactors were loaded in a platform shaker (Innova 2300, New Brunswick, CT, USA) with a speed of 140 rpm. One liter glass reactors were used for all tests, which were conducted in a 37 °C incubation room. For each reactor, a 5 L Tedlar gas bag (CEL Scientific, Santa Fe Springs, CA, USA) was attached to collect biogas every 2–4 days during the 50 day experimental period.

2.3. Analytical methods

The volume of biogas was measured with a drum-type gas meter (Ritter, Bochum, Germany) and its composition (CH₄, CO₂, N₂, and O₂) was analyzed with a gas chromatograph (Agilent, HP 6890, Wilmington, DE, USA) equipped with a 30 m × 0.53 mm × 10 μm Rt[®]-Alumina Bond/KCl deactivation column and a thermal conductivity detector. Helium gas was used as a carrier gas at a flow rate of 5.2 mL/min. The temperature of the detector was maintained at 200 °C, while the initial temperature of the oven was 40 °C and then increased to 60 °C within 1 min.

TS, VS, pH, and alkalinity of digesting materials were measured based on the Standard Methods Examination of Water and Wastewater ([APHA, 2005](#)). Specifically, a 5 g sample was diluted with 50 mL DI water, then the pH and alkalinity was measured using an auto-titrating pH meter (Mettler Toledo, DL22 Food & Beverage Analyzer, Columbus, OH, USA). Total carbon and total nitrogen contents were determined using an elemental analyzer (Elementar Vario Max CNS, Elementar Americas, Mt. Laurel, NJ, USA). Total ammonia nitrogen (TAN) was measured based on a modified distillation and titration method ([ISO 5664, 1984](#)) using 4% boric acid with a AutoKjeldahl Unit K-370 (Buchi Labortechnik AG, Switzerland). Total Kjeldahl nitrogen (TKN) was analyzed based on the Kjeldahl nitrogen method ([ISO 5663, 1984](#)). The difference between TAN and TKN was assumed to be organic nitrogen, based on which, the crude protein content was calculated by multiplying by a factor of 6.25 ([Hattingh et al., 1967](#)). Volatile fatty acids (VFAs), which include propionic, acetic, isovaleric, butyric, isobutyric, and valeric acids, were analyzed using a gas chromatograph (Shimadzu, 2010PLUS, Columbia, MD, USA) equipped with a 30 m × 0.32 mm × 0.5 μm Stabilwax[®] polar phase column and a flame ionization detector, according to a method described previously ([Shi et al., 2013](#)). Extractives in feedstocks were measured based on the NREL Laboratory Analytical Procedure ([Sluiter et al., 2008](#)) using a Dionex ASE 300 extraction system (Thermo Scientific, Sunnyvale, CA), while extractive-free samples were used to determine the structural carbohydrates. Monomeric sugars (glucose, xylose, galactose, arabinose, and mannose) were analyzed using a high-performance liquid chromatograph (Shimadzu, LC-20AB, Columbia, MD, USA) equipped with a Biorad Aminex HPX-87P column and a refractive index detector. HPLC grade water was used as the mobile phase at a flow rate of 0.3 mL/min, and temperatures of the column and detector were maintained at 60 °C and 55 °C, respectively.

2.4. Theoretical methane contribution of each organic component

Previously reported theoretical methane potentials of organic components, which assumed methane potential of 415 and 496 L/kg-VS for carbohydrates (cellulose and hemicellulose) and protein, respectively, were used in this study ([Angelidaki and](#)

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