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Enhancing anaerobic digestion of food waste through biochemical methane potential assays at different substrate: inoculum ratios

Shakira R. Hobbs^{a,*}, Amy E. Landis^a, Bruce E. Rittmann^{b,c}, Michelle N. Young^{b,c}, Prathap Parameswaran^d

^a Institute for Sustainability, Glenn Department of Civil Engineering, Clemson University, Clemson, SC 29634, USA

^b Biodesign Swette Center for Environmental Biotechnology, Arizona State University, Tempe, AZ 85271-5701, USA

^c School of Sustainable Engineering and the Built Environment, Arizona State University, Tempe, AZ 85271-5701, USA

^d Department of Civil Engineering, Kansas State University, Manhattan, KS 66506, USA

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ABSTRACT

Food waste has a high energy potential that can be converted into useful energy in the form of methane via anaerobic digestion. Biochemical Methane Potential assays (BMPs) were conducted to quantify the impacts on methane production of different ratios of food waste. Anaerobic digester sludge (ADS) was used as the inoculum, and BMPs were performed at food waste:inoculum ratios of 0.42, 1.42, and 3.0 g chemical oxygen demand/g volatile solids (VS). The 1.42 ratio had the highest CH₄-COD recovery: 90% of the initial total chemical oxygen demand (TCOD) was from food waste, followed by ratios 0.42 and 3.0 at 69% and 57%, respectively. Addition of food waste above 0.42 caused a lag time for CH₄ production that increased with higher ratios, which highlighted the negative impacts of overloading with food waste. The Gompertz equation was able to represent the results well, and it gave lag times of 0, 3.6 and 30 days and maximum methane productions of 370, 910, and 1950 mL for ratios 0.42, 1.42 and 3.0, respectively. While ratio 3.0 endured a long lag phase and low VSS destruction, ratio 1.42 achieved satisfactory results for all performance criteria. These results provide practical guidance on food-waste-to-inoculum ratios that can lead to optimizing methanogenic yield.

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1. Introduction

Food waste is the largest constituent to municipal solid waste, comprising 21% of waste in landfills by weight in the U.S. in 2012 (EPA, 2014). Landfilling food waste may result in significant greenhouse gas emissions from landfills, since food waste accounts for 13% of methane emissions in landfills (EPA, 2015). The emission of greenhouse gases from food waste has led some states, such as Massachusetts, to set limits on the amount of food waste that can go to landfills (RecylingWorks Massachusetts, 2014). A corollary drawback of landfilling food waste is that its energy value is lost in proportion to the fugitive emissions that contribute to greenhouse gases.

An alternative is to anaerobically digest the food wastes and collect the produced methane. Traditionally, anaerobic digestion (AD) facilities handle organic solids from municipal wastewater treatment plants and farms, and more than 180 anaerobic digester facilities currently operate in the U.S. (EREF, 2015). Some of these facilities recently began adding food waste to the AD input. Food

* Corresponding author at: 112 Lowry Hall, Clemson, SC 29635, US. *E-mail address:* shakirh@clemson.edu (S.R. Hobbs).

http://dx.doi.org/10.1016/j.wasman.2017.06.029 0956-053X/© 2017 Elsevier Ltd. All rights reserved. waste can be an excellent candidate for AD due to its high energy and moisture contents (Cirne et al., 2007; Levis and Barlaz, 2011; Moriarty, 2013). The carbohydrate, protein, and lipid fractions of food waste can be fermented to long-chain fatty acids (LCFAs) and volatile fatty acids (VFAs) that are then converted into acetate and hydrogen gas, the substrates needed by methanogens.

Digesting food waste alone can inhibit methanogenesis. A high risk is that LCFAs and VFAs are produced faster than they can be consumed. Unless the alkalinity is high, this acid accumulation will cause a drop in pH that inactivates methanogens, which function well only within a near-neutral pH range (Buyukkamaci and Filibeli, 2004). The result is a "pickled" digester that accumulates VFAs and H₂, but has minimal chemical oxygen demand (COD) stabilization to CH₄.

A promising strategy is to co-digest food waste with municipal sludge (Elbeshbishy et al., 2012; Liu et al., 2009; Neves et al., 2004). The key to success is a good ratio of food waste to methanogenic biomass. Elbeshbishy et al. (2012) investigated the impacts of the ratio of food waste to inoculum volatile solid (VS) in batch tests. With the pH held constant at 7, CH₄ production increased as the ratio of food waste to methanogenic inoculum increased. However, artificially maintaining a constant pH may not be realistic, and no

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studies have evaluated co-digestion of food waste without externally controlled pH. The ratio of food waste to inoculum will affect the potential to accumulate VFAs, and it also will affect the pHbuffering capacity (Vavilin et al., 2008). Poor understanding of the many impacts could lead to digester upsets (Owen et al., 1979; Rittmann and McCarty, 2001).

The objective of this study was to assess methane production for a range of relevant ratios of food waste to methanogenic biomass. We utilized batch Biochemical Methane Potential (BMP) assays and tested three ratios of food-waste COD to VS of an inoculum of anaerobic digester sludge (ADS). To provide proof of concept and identify food-waste-to-ADS-VS ratios that are promising for further analysis, we measured total chemical oxygen demand (TCOD), semi-soluble chemical oxygen demand (SSCOD), total solids (TS), VS, and pH at the start and end of BMP assays. Other parameters important to AD and methane production were estimated via bicarbonate alkalinity calculations and the Gompertz equation (Lay et al., 1996) for estimating lag times and maximum methane production. Our results provide guidance on ratios needed to sustain good performance by overcoming low-pH inhibition while maintaining good methanogenic yield.

2. Materials and methods

2.1. Food waste recipe and anaerobic digested sludge

The food waste recipe was developed based on weekly food scrap collections at the University of Missouri campus dining operations, as outlined in Costello et al. (2015). The ingredients for the food waste recipe were purchased from a local Wal-Mart food center (Table S1, Supplemental Material). The food waste was prepared by mixing the whole food scraps first by hand, followed by grinding food scraps with 100 mL of water in a food processor (Black and Decker model FP1140BD, USA; 450-Watts) for 10 min on setting 2, which resulted in a paste. The food waste paste was blended (model Black and Decker BL1120SG, USA; 550-Watts) with 200-mL of water for 10 min on setting 4 to create a food waste slurry concentration of 110 g of food waste/L. The AD inoculum for the BMP test was obtained from Mesa Northwest Water Reclamation Plant in Mesa, Arizona, which employs an anoxic-oxic (A/O) process for wastewater treatment. Approximately 60% the primary clarifier solids are diverted to an anaerobic digester, which has a hydraulic retention time ranging of 15-30 days.

2.2. Biochemical methane potential tests/experimental design

BMP tests were performed to determine the amount of CH₄ and H₂ produced from three different COD-to-VS ratios that were based on previous studies with ADS (Angelidaki et al., 2009; Elbeshbishy et al., 2012; Lisboa and Lansing, 2013; Owen et al., 1979): 0.42, 1.42, and 3.0 g COD food waste/g VS ADS. Negative controls (i.e., ADS in basal media without electron donor) were prepared for each ratio, and the methane produced by the controls was subtracted from the total CH₄ on a proportional basis to compute the methane formation from the food waste alone at the end of the BMP assays. The basal medium is described in Supplemental Material. The negative controls did not have any inhibition by low pH, but the food waste BMPs lowered pH and led to pH inhibition at different stages during the BMP test. Thus, we could not do a control subtraction until pH inhibition had been relieved, which occurred by the end of BMP tests in all cases. Therefore, we eliminated the impacts of differential pH inhibition by performing one-time subtraction of the gas production by the negative controls only at the end of the test (day 70). Duplicate positive controls (i.e., ADS with 30 mM acetate as a readily biodegradable electron donor) were set up to ensure that the inoculum was active in methanogenesis and verify the COD conversion to CH_4 .

For each ratio of COD food waste to VS ADS, 120-mL of food waste and ADS mixture was added to 200-mL serum bottles along with 60-mL of deionized water. All ratio bottles were prepared in triplicate. Table 1 shows the volumes of each component used for each experiment. All bottles were sparged with ultra-high-purity N₂ for 10 min to ensure anaerobic conditions. Each serum bottle was sealed with a butyl rubber septum and crimped aluminum cap and placed in an incubated shaker table operated at 180 rpm and a temperature of 37 ± 1 °C. Experiments continued until the daily gas production was <1% of the cumulative gas production, which is discussed further in results (Koch et al., 2015; VDI 4630, 2006).

2.3. Chemical analyses

All analytical tests were performed in triplicate. COD and solids analyses were performed on the food waste, ADS, and initial and final mixtures for all BMP ratios. TCOD and SSCOD, samples filtered through 1.2- μ m glass microfiber filters (Whatman 1822-047 GF/ C)) were assayed using HACH HR COD kits (TNT 821, 20– 1500 mg/L). TS and VS were determined according to *Standard Methods* (APHA, 2012).

pH values were measured using a Cole Parmer pH meter (Vernon Hills, USA). Ammonia nitrogen (NH₃-N) was assayed with HACH kits (TNT832), which had a detection range 2–47 mgNH₃-N/L. Total alkalinity was assayed with HACH kits (TNT870), which had a detection range of 25–400 mgCaCO₃/L. Colorimetric results from all HACH kits were measured using a HACH 2800 spectrophotometer.

2.4. Methane and hydrogen in the biogas

Over a 70-day period, biogas production, i.e., changes in headspace volume at one atmosphere, was measured with a gas-tight glass frictionless syringe (Perfektum, NY). CH₄ and H₂ contents were analyzed using a GC-2010 gas chromatograph (Shimadzu, Japan) equipped with a thermal conductivity detector (TCD) and Carboxen-1010 PLOT capillary column (30 m, Sigma-Aldrich). The TCD was operated with an inlet temperature of 150 °C, a detector temperature of 220 °C, and a current of 41 mA, and argon as carrier gas. Gas-composition analysis involved a temperature program that began at 80 °C for 3 min and was followed by an increase in temperature of 50 °C every minute until 155 °C is reached, giving a total run time of 4.50 min. Methane and hydrogen gas volumes were calculated by multiplying the measured gas composition by the total biogas volume. Electron-equivalent energy recovery (as equivalent COD) was calculated for CH₄ and H₂ according to:

$$1 \text{ mL CH}_4 \text{ gas} = \frac{L}{10^3 \text{ ml}} \cdot \frac{1 \text{ molCH}_4}{22.4 \text{ L}} \cdot \frac{273 \text{ K}}{313 \text{ K}} \cdot \frac{8 \text{ e}^-\text{eq}}{\text{mol CH}_4} \cdot \frac{8 \text{ g COD}}{\text{e}^-\text{eq}} \cdot \frac{10^3 \text{ mg}}{g}$$
$$= 2.52 \text{ mg COD} \tag{1}$$

$$1 \text{ mL } H_2 \text{ gas} = \frac{L}{10^3 \text{ ml}} \cdot \frac{1 \text{ mol } \text{CH}_4}{22.4 \text{ L}} \cdot \frac{273 \text{ K}}{313 \text{ K}} \cdot \frac{2 \text{ e}^-\text{eq}}{\text{mol}H_2} \cdot \frac{8 \text{ gCOD}}{\text{e}^-\text{eq}} \cdot \frac{10^3 \text{ mg}}{g}$$
$$= 0.62 \text{ mg } \text{COD}$$
(2)

2.5. Bicarbonate alkalinity estimation and total alkalinity measurement

The concentration of bicarbonate alkalinity was computed from the final pH and the final CO_2 content in the headspace for each BMP bottle. Eq. (3) was used to estimate the bicarbonate alkalinity:

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