ABSTRACT

BAO, LELE. Evaluating and Predicting the Ultimate Biochemical Methane Potential for Solid Waste from Short-Term Monitoring Data. (Under the direction of Dr. Morton A. Barlaz).

The biochemical methane potential (BMP) test is considered to be a relatively reliable and simple procedure to evaluate methane production of a substrate under anaerobic conditions. The BMP test can be used to provide methane production information for landfill gas recovery projects, to measure the biodegradability of materials that claim to be degradable under anaerobic conditions, and to evaluate the potential toxicity of various materials and leachate. In previous research, the incubation time for BMP tests was often about 100 days and methane production at the end of tests was considered as the ultimate yield. The objectives of this research were to: 1) determine the effect of alternate inocula on methane production, 2) evaluatethe time required for different sample types (municipal solid waste (MSW), green waste, wood and paper) to reach their ultimate methane yield, and 3) to determine whether short-term monitoring data (less than 120 days) can be used to predict the ultimate BMP for the four sample types.

BMP tests were conducted in 160-mL serum bottles incubated at 37°C. To date, samples have been incubated for more than 600 days.Results showed that the BMP measured at 90 days was about 26.1% and 20.7% (median value) greater using anaerobic sewage sludge and leachate as inocula relative to a laboratory-derived culture. In long-term tests with the laboratory culture inoculum, a relationship between (C+H)/L ratio and BMP was observed in MSW and green waste samples (R^2=0.93 and 0.94, respectively).Shorter incubation times (less than 120 days) can be used to estimate the total methane yields, by using BMP_t/BMP

(methane yield in first t days over experimental total yield) and/or first-order decay model methods. When using BMP_t/BMP method, recommended incubation times to obtain sufficient data to extrapolate total methane production for MSW, green waste, wood and paper are 30, 60, 120 and 60 days, respectively. Using the first-order decay model method, the required monitoring times are 150 days for green waste and 120 days for MSW, wood and paper.

Evaluating and Predicting the Ultimate Biochemical Methane Potential for Solid Waste from Short-Term Monitoring Data

by
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DEDICATION

To my family.

BIOGRAPHY

Lele Bao was born on January 1st, 1989 in Jinhua, China. She has been studying in Environmental Engineering since her freshman year. She finished undergraduate courses at Zhejiang University in three years and came to North Carolina State University for graduate study in 2009. In her spare time, Lele loves travelling, reading and playing tennis. Her hobby is Chinese painting.

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

1. Introduction and Objectives

About 250 million tons of municipal solid waste (MSW) were generated in the US in 2008 and 54% (135 million tons) of this waste was discarded in landfills (EPA, 2008). MSW includes considerable degradable organic matter that can be converted to methane including food, paper, wood and green waste. After MSW is buried in landfills, a series of chemical reactions occur in the landfill. Oxygen is depleted quickly and in the absence of electron acceptors such as nitrate and sulfate (Wang et al, 1994), the final products of MSW decomposition are about 50% methane and 50% carbon dioxide. This is referred to landfill gas.

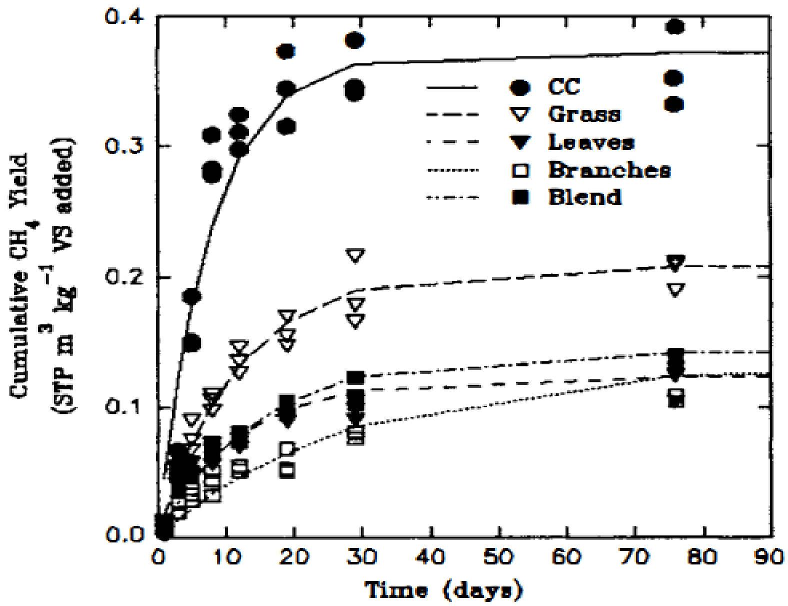
It is reported that thereare currently 551 landfills with gas recovery programs nationwide (EPA, 2011), and there hasbeen an increase in the number of recovery facilities over time. However, many new projects to utilize landfill gas are rejected due to the uncertainty in methane production (Wang et al, 1994). Methane is a greenhouse gas (GHG) that has a global warming potential (GWP) 25 times that of CO_2(IPCC, 2007). Landfill methane emissions are the major GHG emission in the waste sector (IPCC, 2007) and were the second largest human-made source in the United States in 2008 (EPA, 2008). Thus, effective methods to estimate methane production in landfills are needed.

There has been an increase in theapplication of biochemical methane potential (BMP) tests to methane production from different organic samples in last 30 years (Owen et al,1979; Shelton and Tiedje, 1984; Bogner, 1990; Owens and Chynoweth, 1993). The BMP test was first described by Owen et al. in 1979 to measure the biodegradability of a range of organic chemicals under anaerobic conditions (Owen et al, 1979). The test was conducted in 100-mL serum bottles inoculated with mesophilic sludge and incubated at 35°C. Gas production was analyzed every 2 or 3 days for a month. The first application of BMP tests on excavated MSW samples was reported byBogner (1990). 25 g of ground sample was tested using soil as the inoculum. Tests were incubated at 35°Cfor more than 200 days. Results suggested that more biodegradation would be obtained at higher water content and higher soil-refuse ratios. Therefore the addition of liquids into landfills would lead to higher gas production rates and faster stabilization of landfills. In research by Owens and Chynoweth (1993), several MSW components including green waste (grass, leaves, branches) and paper (newsprint, office paper, cardboard, food packaging paper.) were tested using mesophilic sewage sludge for 60 to 80 days at 35°C. The results suggested that grass exhibited higher methane yield than a mixture of green waste (grass, leaves and branches). Newspaper had the lowest methane yields compared to other paper (office paper, cardboard, food packaging paper). Other studies to obtain and compare biodegradability of a variety of samples have been reported. For example, Rao et al (1990) carried out experiments on household garbage, Gunaseelan (2003) on fruit and vegetable waste, and Hasen et al (2004) on food waste. In addition to providing data for biodegradability of MSW components and optimizing design and operation of anaerobic systems (e.g. landfills), BMP tests also provide information to enhance the knowledge of biodegradation process under anaerobic conditions. Wang et al (1994) conducted BMP tests study the degradation of cellulose and hemicellulose in MSW samples, using mesophilic sewage sludge at 37°Cuntil completion of methane production (day 43). This experiment was then followed by a cellulose spike on day 43 to examine the extent of cellulose conversion to methane. Results suggested that cellulose plus hemicellulose was not well correlated with the measured BMP of the samples. The explanation for the poor correlation was that cellulose plus hemicelluloses over lignin ratio was very low (0.02 to 0.12) compared to that observed in other literature (e.g. Stinson and Ham, 1995; Wang et al, 2011). Another study using BMP assays reported that it was not chemical inhibition by printing inks but physical unavailability of cellulose due to lignin that resulted in the lack of cellulose decomposition in newsprint (Stinson and Ham, 1995).

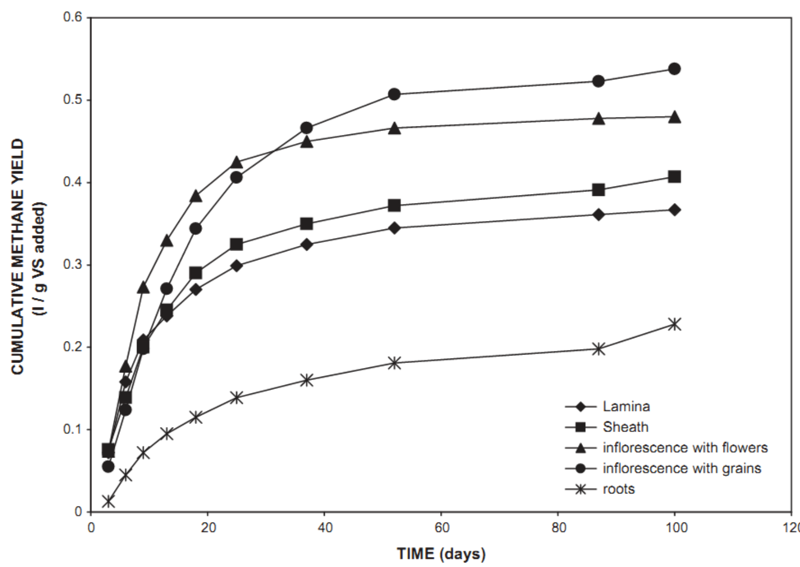
There is no standard protocol to conduct BMP tests that is applicable for general samples/products. The American Society for Testing and Materials (ASTM, 2007) has developed a BMP method for plastic materials. This method requires municipal sewage sludge as theinoculum, but there is no requirement on the incubation time. Moreover,there is limited literature that focuses on factors influencing the BMP tests. Table 1 below summarizes the experimental conditions that have been used to measure the BMP on MSW. Mesophilic sewage sludge is the most common inocula source in BMP tests and samples were often inculcated at 35℃. The incubation durations were usually about 100 days and the incubation time reported was always assumed to represent the ultimate methane yield. However, in many cases in the literature, incremental methane production was reported at the last time point. For example, Figure 1 that was adopted from the research by Owens and Chynoweth (1993) and Gunaseelan (2004) demonstrated that there were still methane productions for all samples during last two analyses. Some studies simply assumed BMP to be equal to ultimate BMP after 100-day incubation (Hashimoto, 1986; Gunaseelan, 2004). While the 100 day incubation time used by many is reasonable, longer periods will detract from the usefulness of the BMP test. Thus, work is needed to determine the appropriate incubation time and to evaluate whether the ultimate methane yield can be predicted from relatively short periods.

Table 1 Summary of experimental conditions in previous application of the BMP test to municipal solid waste

Source	Substrate	Inoculum source	Incubation time	Temperature
Bogner (1990)	MSW samples excavated from a landfill	soil	200 to about 250 days	35℃
Owens & Chynoweth (1993)	Green waste, Paper products	mesophilic sewage sludge	60-80 days	35℃
Wang, Byrd & Barlaz (1994)	MSW samples, cellulose pike on day 43	mesophilic sewage sludge	84 days	37℃
Stinson & Ham (1995)	Newsprint	mesophilic sewage sludge	56 days	35℃
Gunaseelan (2004)	Fruit and vegetable waste	mesophilic sewage sludge	About 100 days	35± 1℃
Hansen et al (2004)	Food waste (fat, oil, protein etc.)	thermophilic biogas plant sludge	About 50 days	55℃
Jeon et al (2007)	Food waste, paper, plastics, wood, textile, rubber, leather etc	mesophilic sewage sludge	Not mentioned	35℃
Neves et al (2008)	Food waste	mesophilic sewage sludge	About 80 days	35℃



(a)



(b)

Figure 1 BMP results inprevious researchesover time

(a) BMP results for different green waste samples(reprinted from Owens and Chynoweth, 1993, cc is the cellulose control) (b) different plant parts of sorghum (reprinted from Gunaseelan, 2004)

1.1 Alternatives for Prediction of Methane Yields

MSW is reported to consist of 40-50 % cellulose, 12% hemicellulose and 10-15% lignin on a dry mass basis; and cellulose and hemicellulose make up 91% of the methane potential in fresh waste (Barlaz, 1990). The methane yield for cellulose and hemicellulose can be calculated from the stoichiometry given below to be 414.8 and 424.2 ml CH_4/dry gm, respectively (Wang et al, 1994):

$$C_nH_aO_b + [n-(a/4)-(b/2)]\ H_2O \rightarrow [(n/2)-(a/8)+(b/4)]\ CO_2 + [(n/2)+(a/8)-(b/4)]\ CH_4$$

However, applicability of a stoichiometric method is limited because it assumes that all cellulose and hemicelluloses degrade. Thus it does not consider influencing factors like bioavailability. In contrast to cellulose and hemicelluloses, lignin is not degraded under anaerobic conditions (Hackett et al, 1977). Additionally, lignin is a major structural polymer found in plants that prevent invasion of microorganisms into plant tissues (Sarkanen and Ludwig,1971). Therefore, lignin makes some cellulose and hemicelluloses physically unavailable during degradation process.

If short term BMP data are available, predictive methods are often based on the assumption that biodegradation process follows a first-order reaction (Owens and Chynoweth, 1993). For example, a nonlinear regression was used to fit to the methane production data in Owens and Chynoweth's research (1993). The regression was performed using Marquardt-Levenberg algorithm available in Sigmaplot (Jandel Scientific, 1989).

1.2 Objectives

The objectives of this research were to: 1) determine the effect of alternate inocula, including a laboratory culture, anaerobic sewage sludge and leachate, on methane production, 2) evaluatethe time required for different sample types (MSW, green waste, wood and paper) to reach their ultimate methane yield, and 3) to determine whether short-term monitoring data (less than 120 days) can be used to predict the ultimate BMP for the four sample types.

2. Materials and Methods

2.1 Experimental Design

In this research, two experiments were designed to evaluatethe BMP methodology. In the biochemical methane potential (BMP) test, the methane potential of a known mass of sample is measured in a 160-mL serum bottles that contains 80 mL of nutrient-rich biological growth medium and 15 mL of inoculum. Five blanks with medium and inoculum only were used to determine the background methane attributable to the inoculum. The first experiment was designed to test the effects of three inocula, a laboratory culture, anaerobic sewage sludge and leachate, on methane production of MSW samples over 90 days. The second experiment was designed to measure methane production over time for a range of MSW components to evaluate the length of time required to reach the ultimate BMP. Tests were conducted with four types of samples (1) municipal solid waste, (2) green waste, (3) wood and (4) paper. Tests were conducted at 37°C and were analyzed for methane yields about every 30 days for the first 180 days, and about every 60 days thereafter to track methane production trends over time.

2.2 Materials

All samples for BMP tests were shredded, dried, ground to a particle size of 1 mm, and then dried at 75 °C for 24 hours. Samples were stored in 16 oz mason jars prior to testing.

2.2.1 Municipal solid waste

Refuse samples were collected through borings from landfill units at the Outer Loop Bioreactor landfill located in Louisville, Kentucky. After excavation, the samples were shipped in 5 gallon buckets to North Carolina State University (NCSU) for analysis. Pieces of metals and textiles were cut into smaller pieces before grinding. Sample details are presented in the Table 2.

Table 2 Sample data summary for municipal solid waste

NCSU ID	Sample Description[a]	Waste Age (Days)	Date Sampled	Date Added to Landfill	Sampling Elevation (ft)
03-262	MSW 1	261	11/15/2003	2/27/2003	507~517
04-13	MSW 2	22	3/20/2004	4/11/2004	n.a.[b]
04-18	MSW 3	76	1/14/04	2/17/2004	n.a.
03-201	MSW 4	1895	11/12/2003	9/4/1998	451~445
04-15	MSW 5	76	3/20/04	2/17/2004	n.a.
03-175	MSW 6	681	11/11/2003	12/30/2001	461~451
03-283	MSW 7	1900	11/17/2003	9/4/1998	464~454
05-202	MSW 8	2903	5/13/2005	6/1/1997	473~483
05-179	MSW 9	3466	5/12/2005	11/15/1995	467~477
05-157	MSW 10	3476	5/11/2005	11/4/1995	440~450
05-159	MSW 11	1623	5/11/2005	11/30/2000	514~524

a. Samples were numbered in the order from highest to lowest based on (C+H)/L ratio.
b. n.a.=not available.

2.2.2 Green waste

The green waste included a mixture of leaves, grass, trimmings, branches and stumps. Samples were obtained from the anaerobic composting digester established at the Yolo County, California (Ramin Yazdani et al, submitted). The digester cell was filled with a mixture of 1,718 Mg of chipped and screened green waste, 118 Mg of aged horse manure as an inoculum, some limestone as buffer and wood chips to protect the liner. Samples were collected from five different layers of the digester during filling fromJune 28 and August 17, 2007.

2.2.3 Wood

Two natural wood samples, red oak (hardwood) and spruce (softwood) were obtained from a local lumberyard. Five types of engineered wood samples were collected from different U.S. manufacturers, which all contained synthetic resins. Plywood (PW) was manufactured from 90% southern yellow pine (softwood). Oriented Strand Board (OSB) was made from 90% aspen and 10% birth (both hardwood), while another OSB was made from 100% southern yellow pine (softwood). Particle Board (PB) and Medium Density Fiberboard (MDF) were made from a mixture of hardwood and softwood (Wang et al, 2011).

2.2.4 Paper

Five samples of newsprint (ONP1 to ONP 5) were provided by Norske Skog as described in Wang et al. (2010). ONP1 was standard newsprint with 60% mechanical softwood, 20% recycled fiber, and 20% semi-chemical hardwood pulp, while ONP2 contained 60% bleached mechanical pulp, 20% recycled fiber and 20% bleached semi-chemical hardwood pulp. ONP 3, ONP 4 and ONP 5 were described as mixtures of virgin softwood and hardwood and some recycled fiber in which both chemical and mechanical pulps were present. Two types of magazine fiber (OMG1 and OMG2) were provided by Australian Paper. OMG 1 was a chemical pulp while OMG 2 was a mechanical pulp. Two samples of copy paper (OFF1 and OFF2) were provided by Fuji Xerox and were made from acacia fiber and eucalyptus fiber, respectively.

2.3 Inocula sources

Leachate was obtained from a 200-L drum of decomposing residential waste that was incubated in the laboratory at 35°C. The lab-derived culture was enriched from a hand-squeezed extract of decomposing residential waste. It has been transferred regularly (about every two weeks) into fresh medium with fresh residential waste as the carbon source for over ten years. Anaerobic sewage sludge was obtained from a mesophilic anaerobic digester at the North DurhamWastewater Treatment Plant.

2.4 BMP procedure

There are three distinct steps in the biochemical methane potential (BMP) test. The first step involves maintaining a lab culture to inoculate BMP assays. This requires preparing maintenance media and transferring the culture into this media every two weeks. The second step is to set up BMP using 160-mL serum bottles and includes media preparation, sample weighing, inoculation and finally incubation at 37°C. The third step is to measure gas production at multiple time points. The bottle headspace was flushed with the same gas as used for medium preparation, 80%/20% N_2/CO_2after each sampling to eliminate carryover methane. After flushing, the headspace was analyzed to confirm that the methane concentration was less than 400ppm (0.04%). Details of the BMP protocol are given in Appendix A.

2.5 Analytical methods

2.5.1 Gas volume and composition

Gas volume was determined by inserting a gas-tight glass syringe equipped with a 23 guage needle through the rubber stopper. Prior to volume measurement, each bottle was shaken manually and a sample was removed for gas analysis.

Gas composition was measured by gas chromatography (GC) (SRI Instruments, Torrance, CA) using a CTR 1 column (Alltech, Deerfield, IL) maintained at 75°C. The valve temperature was kept at 90°C, while the injector and thermal conductivity detector temperatures were both at 100 °C. Helium was used as carrier gas at 88 mL/min. 2.5 mL of gas was injected into the 1 mL sample loop in GC. A flame ionization detector (FID) was used in series to quantify low concentration of methane. The TCD data were used for methane concentrations as low as 5% and the FID was used to quantify methane concentrations between 1 ppmv and 5%. Duplicate analysis was conducted for one out of three bottles to test the reproducibility of sample analysis.

2.5.2 Measurement of cellulose, hemicelluloses and lignin

A known mass of sample was processed through a two-stage acid hydrolysis. The first stage hydrolyzed the sample in 3 mL of 72% (w/v) sulfuric acid for 1 hr at 30°C. After that, 83 mL of deionized water and a fucose internal standard were added to the sample. For the second stage, the sample was autoclaved at 121°C for 1 hr. The sugar hydrolyzates, including arabinose, galactose, glucose, mannose, and xylose, were then analyzed by high-pressure liquid chromatography (HPLC), with an electrochemical detector using a CarboPac PA1 column (4x250 mm)(Wang et al, 2010). The cellulose content was calculated from the mass of glucose and hemicellulose content was converted from the mass of the remaining other sugars. Klason lignin was measured from the weight loss on ignition of remaining solids after acid hydrolysis at 550°C.

2.6 Data Analysis

2.6.1 Calculations for methane production

Methane volume

Methane volume was converted to standard temperature and pressure using the ideal gas law, the conversion of methane volume is derived below.

$$pV = nRT$$

$$\frac{p_{STP} V_{STP}}{T_{STP}} = \frac{p_{measured}\ V_{measured}}{T_{measured}}$$

$$V_{STP} = V_{measured} \ * \frac{273\ K}{temperature\ (°C) + 273\ K}$$

$$* \ \frac{atmosphere\ pressure\ (mmHg)}{760\ mm\ Hg}$$

Where:
$V_{measured} \ = (64\ mL + measured\ volume) * CH_4\%$
64 mL is the headspace in each serum bottle;
CH_4 (%) is measured by GC (v/v).

The BMP value was calculated by diving methane yield by dry sample mass. The equation is shown below.

$$BMP\ \left(\frac{mL\ CH_4}{gram\ sample}\right) = \frac{net\ methane\ yield\ (mL\ at\ STP)}{weight\ of\ sample\ (g)}$$

Where:
Net methane yield = cumulative methane yield – average methane yield of five blanks.

2.6.2 Regression analysis for ultimate methane yield prediction

The yield at the last time point was taken as the ultimate yield and used to evaluate the extent to which data over a shorter period of time could be used to predict the ultimate methane yield. It was assumed that methane production could be described by a first-order reaction. According to de la Cruz (2010), the lab-scale decay rates (k) can be calculated from individual methane production data. Modifications were done to fit the regression analysis with BMP data.

$$\frac{dC}{dt} = -kC$$

where:
C = reactive mass of carbon presented in sample (kg)
t = time of incubation, (day)

Since the reactive mass of carbon is converted to CO_2 and CH_4 in the gas phase and by mass balance, the total reactive carbon is unchanged. Therefore, the equation above can also be used in the case of total volume of CO_2 and CH_4 calculation. By integration, the remaining volume of CO_2 and CH_4 that is not converted due to reactive mass of carbon can be calculated as:

$$C_t = C_u * e^{-kt}$$

where:
C_u = the ultimate CO_2 and CH_4 yield when all reactive mass of carbon is converted to gas, mL/g;

C_t = remaining CO_2 and CH_4 potential in sample, mL/g.

A mass balance on reactive mass of carbon can be done to get the CO_2 and CH_4 yield:

$$y_t = C_u - C_t$$

where:
y_t = CO_2 and CH_4 yield at time t, mL/g.
A nonlinear regression can be done using time and corresponding cumulative CO_2 and CH_4 yields to get decay rate k and estimated ultimate yield C_u. Residual methane potential should be approximately zero when ultimate yield is reached.

$$y_t = C_u \cdot (1 - e^{-kt})$$

$$\frac{dy_t}{dt} = k \cdot C_u e^{-kt}$$

$$\frac{dy_t}{dt} = k \cdot (C_u - y_t)$$

$$Residual = k \cdot (C_u - y_t) - \frac{dy_t}{dt}$$

Therefore, first several sets of BMP (yt) and time (t) data are substituted in the following equation to calculate C_u and k.

$$k \cdot C_u - k \cdot y_t - \frac{dy_t}{dt} = 0$$

Thus, estimated ultimate methane yield can be determined as half of C_u, assuming that 50% of the C is converted to CH_4 and 50% is converted to CO_2.

3. Results and Discussions

3.1 Effect of different inocula on methane production

Ten excavated municipal solid waste samples were tested in BMP assays using three inocula: a lab-derived culture, leachate, and anaerobic sewage sludge. Results are shown in Table 3 and Figure 2. Note that methane measurements were made on days 93, 94 and 89 for the lab culture, leachate and sludge inocula, respectively. However the effects of such time differences on cumulative methane yields were comparatively small as the methane production rates were low in later days compared to earlier days.

The BMP results werecompared using paired t-tests at a 95% confidence level. Results showed that sludge ($p = 1\times10^{-6}$) and leachate ($p = 6\times10^{-7}$) inocula were statistically different from lab culture with respect to cumulative methane yields. Sludge and leachate-inoculated sets had an average of 31.3% (range=4.0% to 66.0%; median=26.1%) and 25.9% (range=7.7%to60.0%; median=20.7%) higher methane yields than the lab culture, respectively. There was no statistically significant difference between the sludge and leachate inocula (p=0.15), with an average percent difference of 2.1% (range=-9.4%to16.2%; median=1.0%).One explanation for the greater methane yields in the sludge and leachate inocula may be that they contained a wider diversity of organisms relative to the lab culture. As presented in Table3, the anaerobic sewage sludge had considerably higher background methane than the leachate and lab culture. However, background methane in the leachate and lab culture was comparable so the higher background methane is not correlated with the observed trends amongst the inocula.

Table 3 BMP results for three inocula on MSW samples

NCSU ID	Sample Description	Lab Culture (mL CH_4/g)[a]	Sludge (mL CH_4/g)[a]	Leachate (mL CH_4/g)[a]
03-175	1	70.3 (10.5)	75.1 (4.0)	76.3 (8.0)
03-201	2	119.1 (2.6)	131.4 (3.0)	137.0 (3.1)
03-283	3	57.9 (5.8)	82.0 (5.7)	78.5 (4.1)
04-13	4	120.8 (6.9)	127.9 (10.5)	139.3 (1.5)
04-15	5	129.3 (3.3)	134.5 (0.1)	139.1 (3.5)
04-18	6	134.8 (4.6)	147.4 (5.3)	150.3 (4.4)
05-157	7	4.9 (0.2)	8.2 (0.3)	7.9 (2.0)
05-159	8	9.8 (1.3)	15.5 (0.2)	14.0 (1.4)
05-179	9	11.4 (0.5)	16.7 (1.0)	15.0 (0.2)
05-202	10	8.6 (0.9)	13.0 (0.5)	10.7 (1.4)
n.a.[b]	Blanks	7.4(0.2)	33.2(0.8)	6.9(0.1)

a. Methane measurements were made on days 93, 94 and 89 for the lab culture, sludge and leachate, respectively. Reported BMP values are the average of triplicates and have been corrected for background methane due to the inoculum. The standard deviation is given in parentheses.

b. n.a.=not applicable. The unit of blanks is mL CH_4 per bottle.

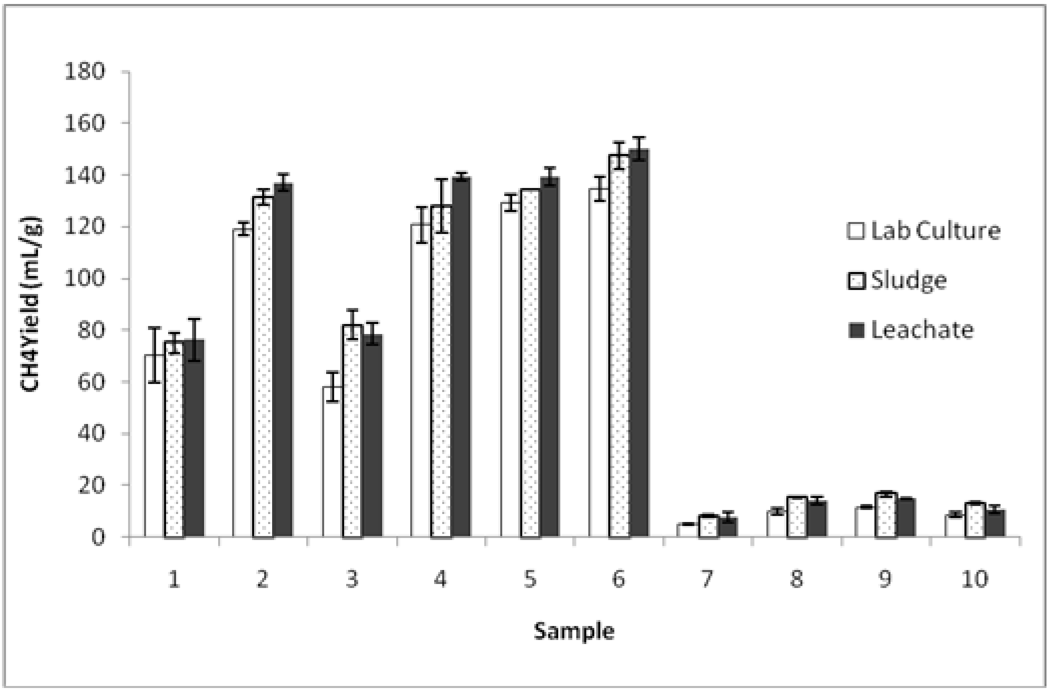


Figure 2 BMP results for three inocula on MSW samples

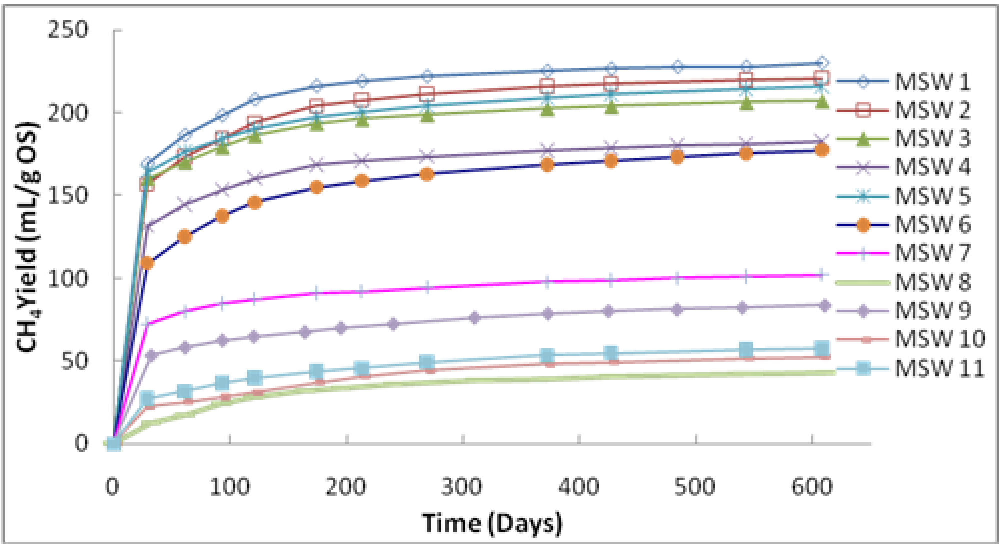
Moreno-Andrade and Buitron (2004) also reported that different sources of inocula could result in different extents of biodegradation and suggested this was because of different levels of microbial population and diversity. A recent study evaluating BMP results among 19 different laboratories from 11 countries on four substrates (starch, cellulose, gelatin and mug bean) was conducted (Raposo et al, 2011). The objective of this study was to investigate influencing factors (inoculum, substrate characteristics and experimental conditions) on BMP test by comparing BMP results on the same four substrates. Each participating laboratories received a full set of samples and basic guidelines on BMP procedures. They were allowed to freely select different inocula and the study included results for 12 mesophilic sludges from different WWTPs, 2 biowaste, 2 manure, 2 brewery sources and 5 industrial process waste WWTP sludges. However, the authors concluded that there was no significant differencein BMP values amongst the inocula. The relative standard deviation (RSD) varied from 15 to 37% for the four substrates tested in this interlaboratory study in which a variety of inocula were used (mainly sludge). By comparison, in this study, the RSD ranged from 3.6 to 26.5% for the 10 samples. The RSD was calculated for each sample on the three inocula to get this range. This suggests that there is still a possibility that the difference in BMP values might due to variations instead of inocula effects.

3.2 Effect of different waste types on methane production

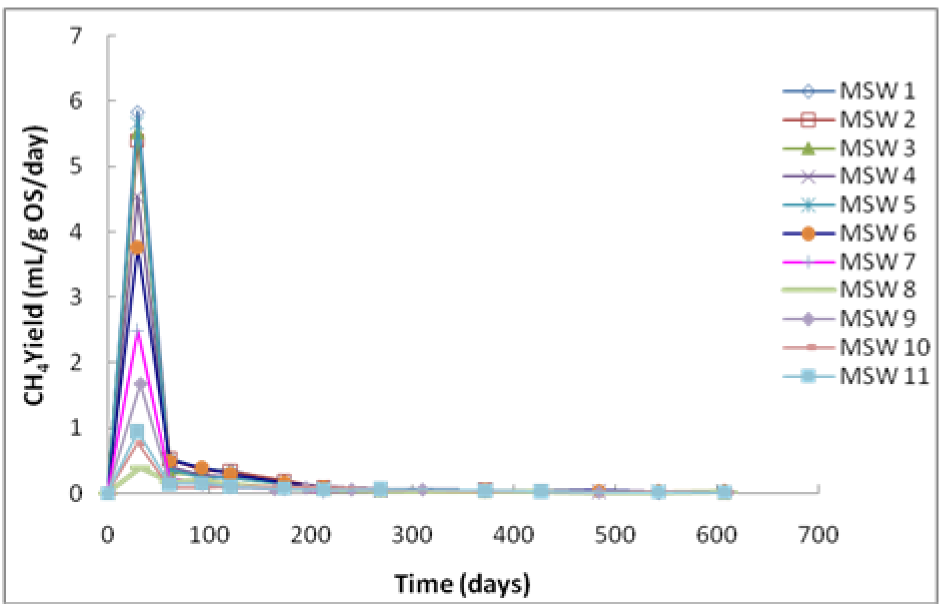
3.2.1 Municipal Solid Waste

Cumulative methane productionfor 11 MSW samples is presented in Figure3.All methane production data were converted to mL CH_4 at STP per gram of organic solids. Samples were numbered from highest to lowest based on (C+H)/L ratio (sum of cellulose and hemicellulose divided by lignin). Chemical composition and methane production data are presented in Table4.

Figure 3 demonstrates that all MSW samples had an initial rapid methane production phase during the first 30 days after which the rate decreased. Of course, the exact shape of the curves in Figure 3 is controlled to some extent by the sampling times. A wide range of methane production (43.0 to 230.0 mL CH_4/g OS) was observed among different MSW samples. Methane production rates in the first 30 days ranged from ~0.4 to 5.8 CH_4/(g OS-day) and dropped quickly to 0.09 to 0.5 CH_4/(g OS- day) from days 30 to 60. The average production rates for measurements after 300 days (days 372 to 608 for MSW 17, 10 to 11 and 310 to 611 for MSW 8 to 9) approached zero (range from 0.02 to 0.04 CH_4/(g OS- day)).



(a)



(b)

Figure 3 BMP results for MSW samples over time. (a) cumulative methane yield. (b) methane production rate.

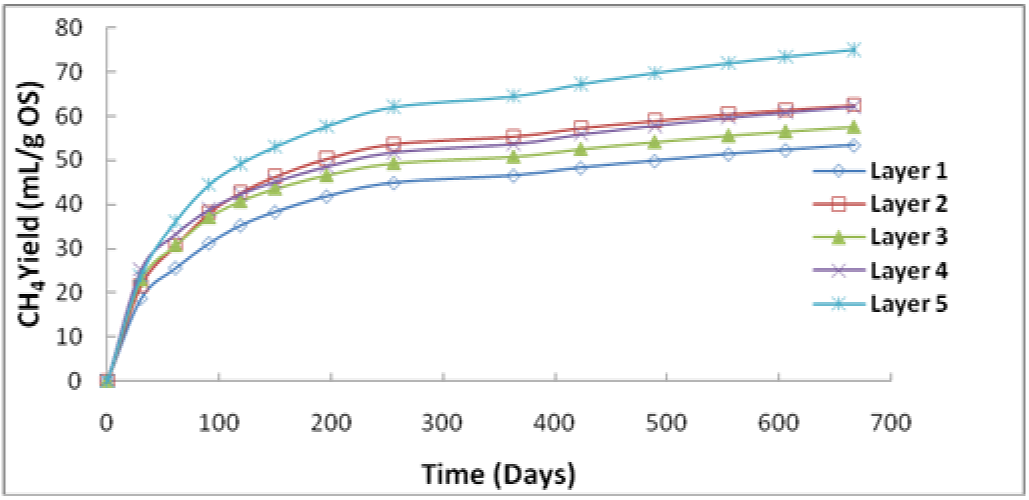
Table 4 Chemical composition and methane yield data for MSW samples

NCSU ID	Sample Description	Waste Age (days)	Days of Incubation[a]	Cellulose (%)[b]	Hemi-cellulose (%)[b]	Lignin (%)[b]	C+H/L[c]	Organic Solids[b] (%)	BMP (mL CH_4/g)[d]	BMP(OS) (mL CH_4/g OS)[e]	Theoretical Methane Yield (mLCH_4/g)[f]	Extent of Conversion (%)[g]
03-262	MSW 1	261	608	45.8	11.2	15.7	3.6	79.4	182.6 (20.2)	230.0	237.6	76.9
04-13	MSW 2	22	608	37.1	9.1	14.6	3.2	65.5	144.6 (4.1)	220.8	192.7	75.0
04-18	MSW 3	76	608	39.8	9.0	15.8	3.1	75.2	155.7 (4.9)	207.2	203.5	76.5
03-201	MSW 4	1895	608	43.0	9.7	20.3	2.6	77.7	142.0 (10.7)	182.8	219.3	64.8
04-15	MSW 5	76	608	33.0	7.4	15.6	2.6	70.3	151.7 (7.1)	215.9	168.2	90.2
03-175	MSW 6	681	608	26.4	7.4	19.2	1.8	51.1	90.8 (8.0)	177.5	140.9	64.4
03-283	MSW 7	1900	608	25.5	4.8	22.5	1.3	68.4	69.9 (7.8)	102.2	126.1	55.4
05-202	MSW 8	2903	611	8.5	3.0	18.3	0.6	35.3	15.2 (1.5)	43.1	48.1	19.1
05-179	MSW 9	3466	611	3.9	1.2	8.1	0.6	18.3	15.3 (0.9)	83.8	21.1	72.5
05-157	MSW 10	3476	608	5.0	1.6	10.7	0.6	17.5	9.2 (1.5)	52.5	27.7	54.8
05-159	MSW 11	1623	608	5.9	1.9	17.8	0.4	26.9	15.5 (2.4)	57.7	32.3	48.0

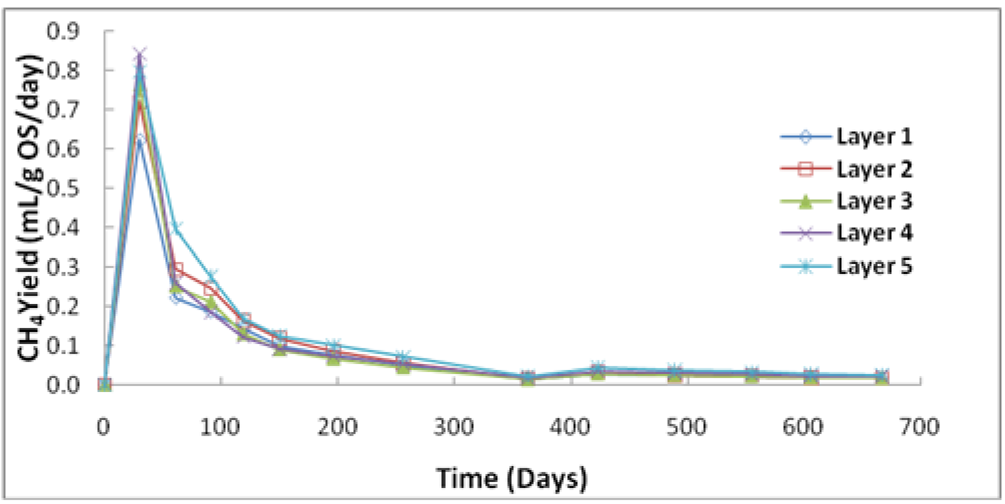
a. The last measurement day is different because samples were tested in separate sets. MSW samples continue to make methane.
b. % of dry weight. Reported values are the average of duplicates and the relative percent deviations were below 10%.
c. C+H/L is (cellulose+hemicellulose)/lignin ratio.
d. Reported methane production data are the average of triplicates and have been corrected for the background methane due to the inoclum. Standard deviations are given in brackets.
e. BMP(OS) is the methane yield normalized to the organic solids content.
f. Theoretical methane yield is determined by stoichiometric conversion of cellulose and hemicellulose to methane.
g. Extent of conversion is the experimental methane divided by theoretical methane yield.

3.2.2 Green Waste

The cumulative methane yields for five green waste samples ranged from 53.5 to 75.1 mL CH_4/g OS. Layers 1 and 2 (6.8% old horse manure) and layers 3 to 5 (no manure) resulted in similar methane production, which shows that the 6.8% horse manure is insignificant in its contribution to the total methane yield. In general, methane production rates in the first 30 days ranged from 0.6 to 0.8 CH_4/(g OS- day) and declined gradually to 0.2 to 0.4 CH_4/(g OS- day) from days 30 to 60. The green waste samples demonstrated a linear increase in methane yields from day 363 to day 667, with an average methane production rate of 0.02 mL CH_4/(g OS- day) (st.dev=0.007, range from 0.02 to 0.03 mL CH_4/(g OS- day)).



(a)



(b)

Figure 4 BMP results for green waste over time. (a) cumulative methane yield. (b) methane production rate.

Table 5 Chemical composition and methane yield data for green waste

NCSU ID	Sample Description[a]	Days of Incubation[b]	Cellulose (%)	Hemi-cellulose (%)	Lignin (%)	C+H/L[c]	Organic Solids (%)	BMP (mL CH_4/g)[d]	BMP(OS) (mL CH_4/g OS)	Theoretical Methane Yield (mLCH_4/g)[e]	Extent of Conversion (%)[f]
09-543	Layer 1	667	5.0	1.9	21.7	0.3	37.2	19.9 (0.3)	53.5	28.8	69.0
09-544	Layer 2	667	5.9	1.8	19.1	0.4	36.9	23.1 (1.9)	62.5	32.2	71.6
09-545	Layer 3	667	4.6	1.8	20.9	0.3	32.9	18.9 (0.5)	57.5	26.8	70.5
09-546	Layer 4	667	7.0	2.8	24.8	0.4	41.0	25.5 (0.5)	62.2	40.7	62.7
09-547	Layer 5	667	9.1	3.6	20.0	0.6	37.0	27.7 (1.0)	75.1	53.0	52.4

a. Layer 1 and 2 were the bottom layers filled with green waste, horse manure and limestone. Layers 3 to 5 were the upper layers and filled with green waste and limestone only.
b. All samples continue to make methane as of day 667.

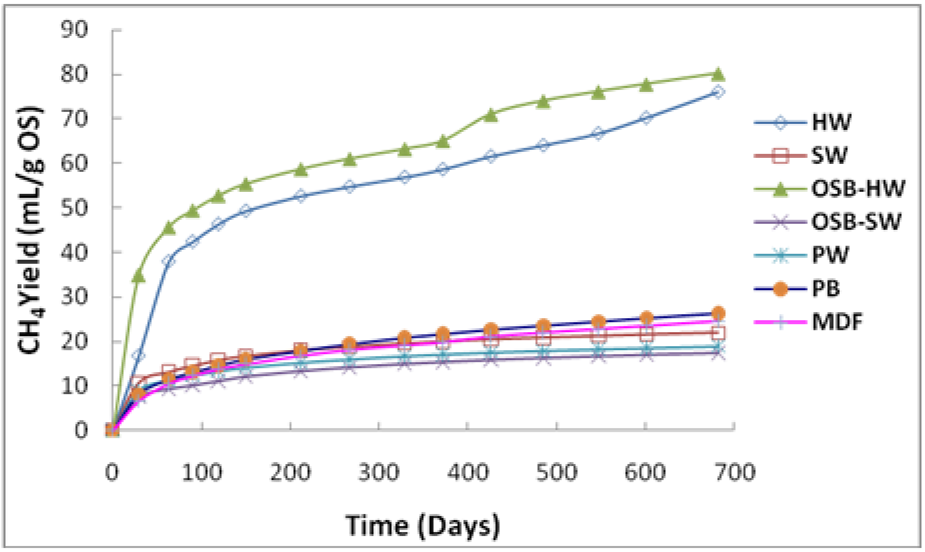
3.2.3 Wood Samples

Continuous methane production has been observed through day 687 for all seven wood samples as presented in Figure 5. The behavior of natural lumber in the form of hardwood (HW) and softwood (SW) is discussed first, followed by presentation of the behavior of engineered woods and a comparison of BMP and reactor methane production data.

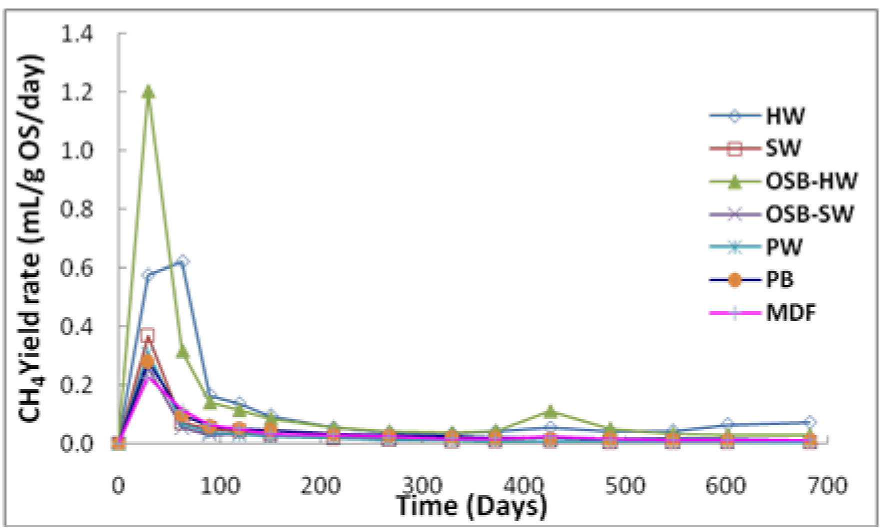
Significant differences in methane yields between HW (76.1 mL CH_4 /g OS) and SW (22.0 mL CH_4 /g OS), and between OSB-HW (80.3 mL CH_4 /g OS) and OSB-SW (17.4 mL CH_4 /g OS) were observed. These trends between HW and SW are consistent with trends measured in reactors studies (Wang et al., 2011). The higher methane yields in the HW were attributed to differences in lignin polymer structures; the lower structural integrity in HW makes it easier for bacteria to access the cellulose and hemicellulose (Wang et al., 2010). HW and OSB-HW had methane production rates of 0.58 and 1.2 mL CH_4/(g OS- day) for first 29 days. HW even demonstrated a slight increase in rate from 0.58 to 0.62 mL CH_4/(g OS- day) between days 29 and 63, while OSB-HW dropped to 0.32 mL CH_4/(g OS- day) . It is surprising to see that they both had an increase in the rates even after 300 days. The methane production rate in SW and OSB-SW was 0.23 to 0.31 mL CH_4/(g OS- day) through day 30, after which it decreased to 0.07 to 0.11 mL CH_4/(g OS- day) from day 30 to 60.

Other engineered woods demonstrated relatively low methane production. PW (mainly SW) produced 18.9 mL CH_4 /g OS; PB and MDF (mixture of HW and SW) had slightly more methane production; 26.4 and 24.6 mL CH_4 /g OS, respectively. One possible explanation is the lower (C+H)/L ratios in engineered wood compared to natural wood (except for OSB-HW). Methane production rates of PW, PB and MDF in first 29 days ranged from 0.2 to 0.3 mL CH_4/(g OS- day) and decreased to 0.07 to 0.1 mL CH_4/(g OS- day) over the next 30 days.

The methane yields of each of the woods were also measured in a reactor study (Wang et al., 2011) and the results are presented in Table 7. With the exception of OSB-HW, reactor methane yields are lower than the BMP results. The results for the OSB-HW are surprising and there is no apparent explanation. Otherwise, similar trends were observed in the BMP and reactor studies as the HW and OSB-HW had much higher yields than the other wood samples.



(a)



(b)

Figure 5 BMP results for wood samples over time. (a) cumulative methane yield. (b) methane production rate.

Table 6 Chemical composition and methane yield data for wood

NCSU ID	Sample Description	Days of Incubation [a]	Cellulose (%)	Hemi-cellulose (%)	Lignin (%)	C+H /L	Organic Solids (%)	BMP (mL CH_4/g)	BMP(OS) (mL CH_4/g OS)	Theoretical Methane Yield (mLCH_4/g)	Extent of Conversion (%)
07-151	HW	682	40.5	19.6	23.5	2.6	99.6	75.8 (0.6)	76.1	250.9	30.2
07-150	SW	682	42.0	20.3	27.4	2.3	98.5	21.6 (0.8)	22.0	260.1	8.3
07-152	OSB-HW	682	42.1	16.8	22.5	2.6	99.0	79.4 (8.0)	80.3	245.8	32.3
08-1876	OSB-SW	682	37.6	17.9	33.6	1.7	98.9	17.2 (0.4)	17.4	232.0	7.4
07-153	PW	682	38.8	17.0	31.4	1.8	96.4	18.3 (0.1)	18.9	232.9	7.8
07-154	PB	682	37.3	16.3	28.2	1.9	98.9	26.1 (0.9)	26.4	223.5	11.7
07-155	MDF	682	34.8	15.2	29.5	1.7	98.6	24.2 (0.6)	24.6	208.8	11.6

a. All samples continue to make methane as of day 682.
b. The lignin content for OSB and PW was not corrected for phenol formaldehyde resin. The reported values likely include about 3% resin.

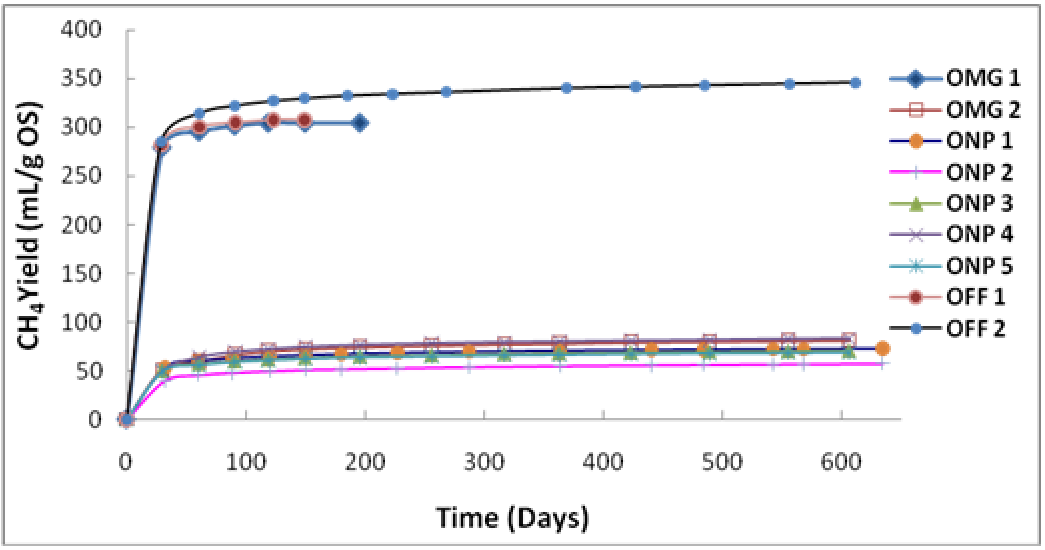
Table 7 Chemical composition and methane yield data for wood

NCSU ID	Sample Description	BMP (mL CH_4/g)	Reactor methane yield (mL CH_4/g)[a]
07-151	HW	75.8 (0.6)	32.4 (11.8)
07-150	SW	21.6 (0.8)	7.5 (0.7)
07-152	OSB-HW	79.4 (8.0)	83.7 (8.1)
08-1876	OSB-SW	17.2 (0.4)	-0.1 (0.2)[b]
07-153	PW	18.3 (0.1)	6.3 (5.0)
07-154	PB	26.1 (0.9)	5.6 (3.3)
07-155	MDF	24.2 (0.6)	4.6 (0.7)

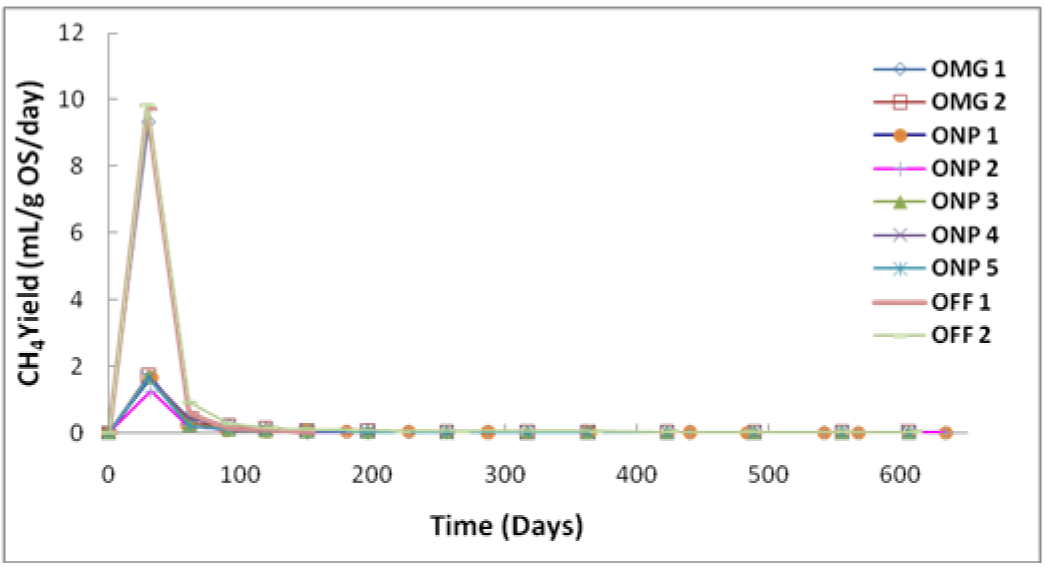
a. The reactor methane yield data are adopted from paper by Wang et al. (2011).
b. The negative number means that the average methane production from OSB-SW was smaller than the average of refuse seed that were used to determine background methane.

3.2.4 Paper Products

All paper samples reached ultimate yields within 650 days, with comparatively high methane production rates and BMP values. Office paper (OFF 1 and OFF 2, both made of chemical pulp) demonstrated the highest methane production (307.8 and 346.2 mL CH_4/g OS, respectively). For magazines, OMG 1 (from chemical pulp) exhibited a higher methane yield than OMG 2 (from mechanical pulp). This is due to the chemical process that removed lignin (Nakamura and Mtui, 2003) and made cellulose and hemicelluloses bioavailable. Newsprint samples (mechanical pulp) had relatively smaller methane yields (57.6 to 83.1 mL CH_4 /g OS). The highest methane production rates in first 30 days in this study were found in paper products, ranging from 9.3 to 9.8 mL CH_4/(g OS- day) for those made of chemical pulp (OFF1, OFF2 and OMG 1) and from 1.2 to 1.7 mL CH_4/(g OS- day) for those made of mechanical pulp (ONP1 to 5 and OMG 2).



(a)



(b)

Figure 6BMP results for paper products over time. (a) cumulative methane yield. (b) methane production rate

Table 8 Chemical composition and methane yield data for paper products

NCSU ID	Sample Description	Days of Operation [a]	Cellulose (%)	Hemi-cellulose (%)	Lignin (%)	C+H /L	Organic Solids (%)	BMP (mL CH_4/g)	BMP(OS) (mL CH_4/g OS)	Theoretical Methane Yield (mLCH_4/g)	Extent of Conversion (%)
08-81	OMG 1	196	42.3	7.4	3.3	15.1	55.9	170.4 (5.4)	305.0	206.7	82.4
08-82	OMG 2	606	34.5	11.9	19.6	2.4	71.5	58.2 (13.0)	81.4	193.4	30.1
08-78	ONP 1	634	45.2	18.0	23.9	2.6	96.0	70.0 (3.1)	72.9	263.8	26.5
08-79	ONP 2	634	43.2	17.9	25.5	2.4	95.6	55.1 (2.7)	57.6	255.3	21.6
08-1985	ONP 3	606	45.6	17.1	24.3	2.6	96.6	68.6 (1.7)	71.0	262.0	26.2
08-1986	ONP 4	606	45.2	17.7	24.5	2.6	96.6	80.3 (20.7)	83.1	262.7	30.6
08-1987	ONP 5	606	44.3	16.5	24.1	2.5	96.6	66.3 (2.1)	68.6	253.7	26.1
08-80	OFF 1	149	61.3	11.4	1.7	41.7	77.2	237.5 (8.6)	307.8	302.6	78.5
08-852	OFF 2	611	57.8	12.2	0.2	424.6	77.3	267.6 (14.8)	346.2	291.8	91.7

a. Methane production in all paper samples was complete within 634 days. OMG1 and OFF1 were completed on days 196 and 149 days, respectively.

3.2.5 Discussion

3.3.5.1 Methane production

Table 9 summarizes the BMP results and methane production rates for the four types of samples. Wood and paper products have subtypes that are classified depending on the raw materials used in the manufacturing process. MSW samples had a wide range of BMP values which is expected as these samples were pre-selected to represent a large range in (C+H)/L values. Green waste had lower BMPs and a narrower range. As all five samples represented green waste of the same age, the narrow range could be expected. There is a large range in BMPs for the wood can be attributed to the wood type (HW vs. SW), while the large differences in the paper can be attributed to the presence of chemical vs. mechanical pulp. Note that paper products were the only samples that reached ultimate yields in this study (~ 650 days), which is consistent with the initial objective to explore appropriate incubation times for BMP tests.

Methane production rates for MSW between days 30 and 60 were 9.8% of the rate up to day 30, except for MSW 8. MSW 8 showed a slower decrease in methane rate; the average rate of day 30 to 60 divided by that of day 0 to 30 is 43.8%). For green waste, the day 30 to 60 rate was 38.1% of that in first 30 days. The slowest decline in the methane production rate was in hardwood (HW, OSB-HW), and HW even demonstrated an increase (0.58 to 0.62 mL CH_4/(g OS- day)) in methane production rate after day 547. Paper products showed the fastest biodegradation in first 30 days among four types of substrates studied, regardless of pulp type. MSW, green waste and hardwood (HW, OSB-HW) demonstrated relatively high methane production rates after 300 days (>0.02 mL CH_4/(g OS- day)).

Table 9 Summary of BMP and methane production rates for four types of samples

Type of sample		Cumulative methane yields (mL CH_4/g OS)	Average methane production rate (mL CH_4/(g OS- day))		
			0 to 30 days[a]	30 to 60 days[a]	300 days to present[a]
MSW		43.0~230.0	0.4~5.8	0.09~0.5	0.02~0.04
Green waste		53.5~75.1	0.6~0.8	0.2~0.4	0.02~0.03
Wood	Hardwood (HW, OSB-HW)	76.1~80.3	0.6, 1.2	0.6, 0.3	0.05, 0.05
	Softwood (SW, OSB-SW)	17.4~22.0	0.4, 0.3	0.07, 0.05	0.01, 0.01
	Other engineered wood (mixture of HW+SW)	18.9~26.4	0.2~0.3	0.07~0.1	0.01~0.02
Paper products	Chemical pulp (2 OFF, OMG1)	305.0~346.2	9.3~9.8	0.5~0.9	0~0.03
	Mechanical pulp (5 ONP, OMG2)	57.6~83.1	1.2~1.7	0.2~0.4	0.009~0.014

a. Depending on actual analysis dates, it may not necessarily be exact 30, 60 or 300 days. Details are given in the Appendix C.

3.2.5.2 BMP and (C+H)/L ratio relationships

The C+H/L ratio is typically used as an indicator of biodegradability under anaerobic conditions; since lignin does not degrade anaerobically (Hackett et al, 1977). Results from this study once again confirmed this usage of (C+H)/L ratio for roughly judging biodegradability.

Figure 8 demonstrates the relationship between BMP and (C+H)/L based on each substrate. Overall, a higher (C+H)/L resulted in higher methane yields.The relationship was strongest for MSW and green waste samples, with regression coefficients of 0.93 and 0.94, respectively. For wood samples, (R^2 = 0.76) is lower than that of MSW and green waste. One potential explanation is that wood samples were not as degradable; there were fewer samples with a wider range of properties (i.e., HW vs. SW, lumber vs. wood containing synthetic resins).Similarly, the relationship between BMP and (C+H)/L is not good for paper given the large differences in chemical composition between chemical and mechanical pulps.

In conclusion, (C+H)/L ratio can be roughly used to predict BMPfor MSW and green waste but not for wood and paper.

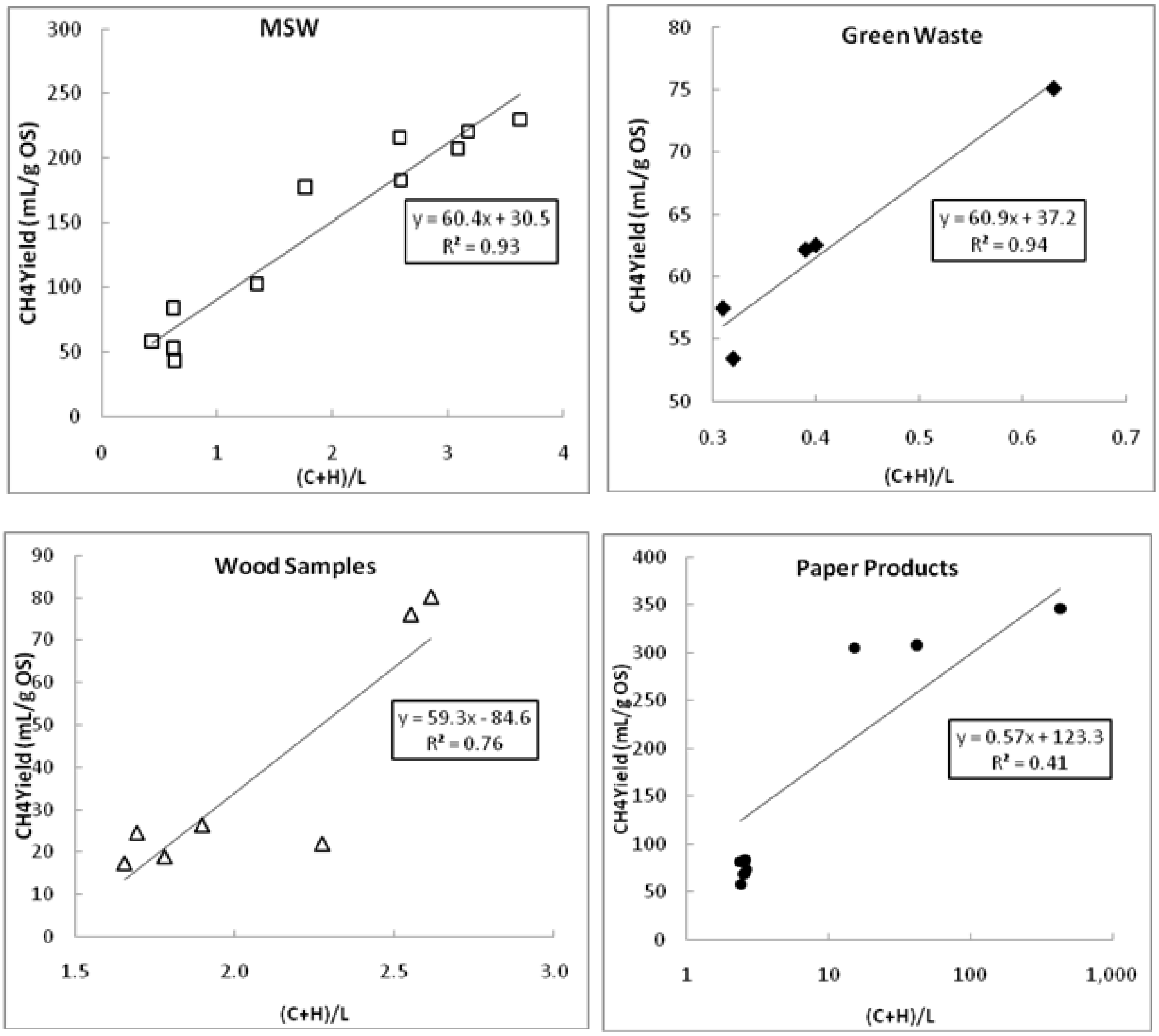


Figure 7 Relationships between BMP and (C+H)/L ratio by each waste type

3.3 Indicators for predicting ultimate methane production

All samples continued to degrade after the first 100 days, which indicates that the traditional 100-day BMP tests are not sufficient to show the cumulative yields and therefore might not be considered as a convincing or satisfying tool to assess biochemical methane potentials. Alternative ways to predict ultimate methane yields in shorter periodsarediscussed in this section.The objective of this section is to explore methods to predict the ultimate BMP by using short term (less than 120 days) BMP data. Two predictive methods are proposed: 1) BMP_t/BMP total method, 2) first-order decay model. BMP_t/BMP total is the methane yield at day t±2 relative to the total yield. For this analysis, the total yield is assumed to be the measured BMP at the last time point, although samples continue to produce methane.

3.3.1 BMP_t/BMP method

The goal of this method is to estimate total methane production by using BMP data for 30 to 90 days as a fraction of the total yield. Figure 9 demonstrates the relationship between (C+H)/L and BMP_t/BMP. Overall, (C+H)/L ratio does not affect BMP_t/BMP ratio in most cases, except that more variability in BMP_t/BMP ratios was observed for MSW samples when (C+H)/L ratios were below 1.The CV (coefficient of variation) was used as a metric to compare predictability of 30, 60 and 90-day data. As presented in Table 10, the CV generally decreases as the time increases in the numerator of BMP_t/BMP. The preferred monitoring time was selected as the time when the CV was below 10%. This occurred for MSW (when (C+H)/L>1), green waste, paper and wood at 30, 60, 120, and 60 days, respectively. More uncertainty was observed when using BMP_t/BMP to predict total methane yield for MSW when (C+H)/L is less than 1; relatively wide ranges were found in BMP30/BMP (0.28 to 0.65) and BMP60/BMP (0.38 to 0.71) ratios.

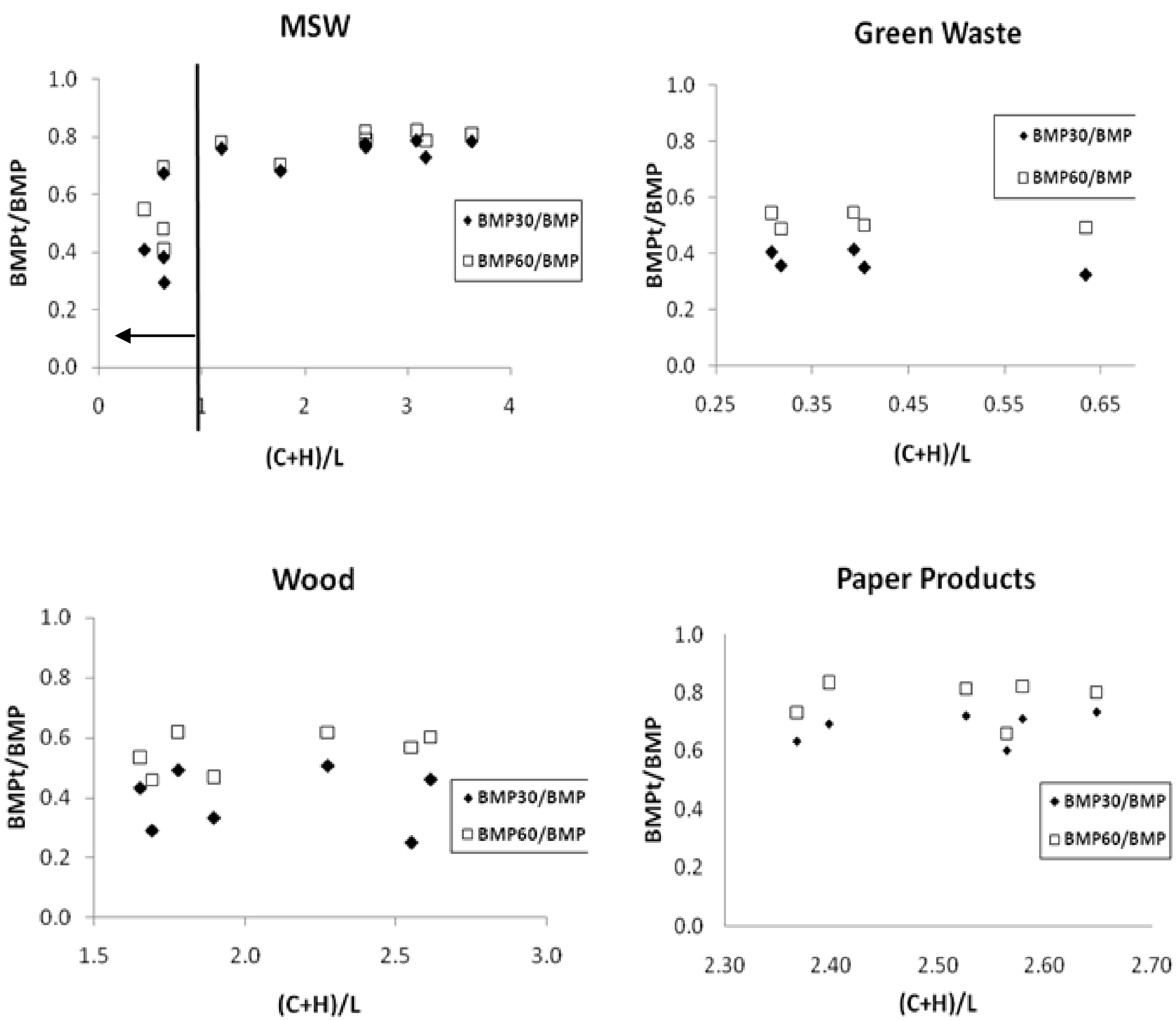


Figure 8Relationships of BMP data and (C+H)/L ratio, the x-axis does not always start from zero is due to the ranges of (C+H)/L ratios differed between four types of samples.

Table 10 Summary of prediction results of BMP_t/BMP method

	BMP30/ BMP[a]	CV[b] (%)	BMP60/ BMP[a]	CV[b] (%)	BMP120/ BMP[a]	CV[b] (%)	Preferable Operation Time for BMP test[c] (days)
MSW	0.62	24.7	0.70	20.9	0.80	13.6	-
MSW (C+H)/L<1	0.46	29.2	0.54	21.9	0.68	11.3	-
MSW (C+H)/L>1	0.72	7.8	0.79	6.1	0.87	3.7	30
Green Waste	0.37	11.0	0.51	6.5	0.69	3.5	60
Wood	0.39	25.1	0.55	12.2	0.67	9.0	120
Paper Products	0.75	15.3	0.85	10.5	0.91	6.3	60

a.BMP_t/BMP = methane yield at day t relative to the total yield at last measurement. Day t refers to a range of t±2 days depending on the actual measurement date. Therefore t may not be exactly day 30, 60 or 120.
b. CV= coefficient of variation, calculated as standard deviation divided by the mean.
c. Preferable operation time t for BMP testis determined when less than about 10% CV value among BMP_t/BMP ratiowas observed.

3.3.2 First-order decay model

The use of a first order decay model to predict ultimate BMPs was also explored. Prediction results vary depending on the number of data points used in the model (summarized in Table11). If using first three measurements (~30, 60, 90 days) to predict total methane production, the results were not satisfying. The predicted ultimate BMP over experimental BMP (BMPu/BMP) ranged from -7.3 to 3.7, 3.0 to 6.0, 1.2 to 3.1 and 1.1 to 3.3 for MSW, green waste, wood and paper products, respectively. When the first four measurements (0 to 120 days) were used in the model, MSW, wood and paper products resulted in good estimates of ultimate yields as presented in Table 11 (average BMPu/BMP = 0.98, 0.94 and 1.00, respectively, excluding MSW 8). MSW 8 is an outlier as the production rate decreased more slowly over the first 120 days relative to the other MSW samples. The relationship between the rate decrease and prediction results is discussed in Appendix B 1.2. For MSW 8, if first 240 days are used, then the prediction is satisfying (1.10 of actual observation). In the case of green waste, predictions using four data points through day 119 were 1.25 times the actual methane yields on average. Using five measurements (through day 150), good estimates were obtained (BMPu/BMP =1.03 st.dev=0.08).

Table 11 Prediction results of first-order decay model

Sample Type	Sample Description	BMP (mL CH_4/g)[a]	BMPu_90 (mL CH_4/g)[a]	BMPu_90/ BMP	BMPu_120 (mL CH_4/g)[a]	BMPu_120/ BMP	BMPu_150 (mL CH_4/g)[a]	BMPu_150 /BMP
MSW	MSW1	182.6	307.2	1.7	188.5	1.0	182.7	1.0
	MSW2	144.6	226.0	1.6	145.8	1.0	144.2	1.0
	MSW3	155.7	574.9	3.7	156.9	1.0	154.4	1.0
	MSW4	142.0	222.5	1.6	141.1	1.0	140.2	1.0
	MSW5	151.7	195.0	1.3	145.4	1.0	144.7	1.0
	MSW6	90.7	238.7	2.6	92.3	1.0	88.8	1.0
	MSW7	69.9	106.2	1.5	65.9	0.9	65.2	0.9
	MSW8	15.2	-110.6	-7.3	43.0	2.8	24.7	1.6
	MSW9	15.3	46.0	3.0	13.5	0.9	13.2	0.9
	MSW10	9.2	-38.2	-4.2	8.1	0.9	11.5	1.2
	MSW11	15.5	-58.4	-3.8	16.0	1.0	15.0	1.0
Green Waste	Layer 1	19.9	119.6	6.0	26.2	1.3	21.5	1.1
	Layer 2	23.0	167.6	7.3	32.3	1.4	25.6	1.1
	Layer 3	18.9	111.8	5.9	23.3	1.2	19.2	1.0
	Layer 4	25.5	77.1	3.0	26.6	1.0	23.4	0.9
	Layer 5	27.7	130.5	4.7	34.4	1.2	27.9	1.0
Wood	HW	75.8	232.7	3.1	81.2	1.1	67.3	0.9
	SW	21.6	36.2	1.7	21.4	1.0	19.7	0.9
	OSB-HW	79.5	99.1	1.2	66.4	0.8	62.2	0.8
	OSB-SW	17.2	20.8	1.2	14.7	0.9	14.8	0.9
	PW	18.3	25.6	1.4	16.9	0.9	15.9	0.9
	PB	26.1	42.6	1.6	23.3	0.9	21.6	0.8
	MDF	24.2	54.8	2.3	24.7	1.0	20.8	0.9

Table 11 Continued

Sample Type	Sample Description	BMP (mL CH_4/g)[a]	BMPu_90 (mL CH_4/g)[a]	BMPu_90/ BMP	BMPu_120 (mL CH_4/g)[a]	BMPu_120/ BMP	BMPu_150 (mL CH_4/g)[a]	BMPu_150 /BMP
Paper Products	OMG 1	170.4	197.4	1.2	175.4	1.0	172.2	1.0
	OMG 2	58.1	189.6	3.3	62.8	1.1	57.2	1.0
	ONP 1	70.0	87.1	1.2	67.6	1.0	65.6	0.9
	ONP 2	55.1	72.1	1.3	52.8	1.0	50.9	0.9
	ONP 3	68.6	87.0	1.3	65.7	1.0	63.8	0.9
	ONP 4	80.3	134.6	1.7	83.5	1.0	78.1	1.0
	ONP 5	66.3	88.6	1.3	64.7	1.0	62.6	0.9
	OFF 1	237.5	264.4	1.1	243.9	1.0	235.8	1.0
	OFF 2	267.6	304.3	1.1	264.5	1.0	258.9	1.0

a. Based on per gram dried sample, instead of organic solids. BMPu is the ultimate BMP predicted by the first order decay model.

Table 12 summarizes the prediction results. The criteria used to determine preferable operation time include: 1) less than 10 % CV, 2) BMP predicted results approach actual observations. Preferable monitoring times to estimate the total yields are 120 days for MSW, paper and wood, and 150 days for green waste.

Table 12 Summary of prediction results of first-order decay model

	BMPu_90 /BMP[a]	CV[b] (%)	BMPu _120/ BMP[a]	CV[b] (%)	BMPu _150/BMP[a]	CV[b] (%)	Preferable Operation Time for BMP test[c] (days)
MSW	0.90	296.9	0.98	5.9	0.99	10.0	120
Green Waste	5.39	29.8	1.25	10.7	1.03	7.3	150
Wood	1.72	45.2	0.94	10.4	0.86	5.6	120
Paper Products	1.65	54.8	1.00	5.5	0.96	4.0	120

a. BMPu=ultimate yield prediction from model;
b. CV= coefficient of variation, calculated as standard deviation divided by the mean.
c. Preferable operation time t for BMP testis determined when the CV is less than 10%.

3.3.3 Comparisons of prediction methods

A comparison of BMP_t/BMP and first-order decay model methods is presented in Table13. BMP_t/BMP required shorter monitoring times than the model prediction (30 to 120 and 120 to 150, respectively). However, the fraction of the total BMP that is actually measured is below 75% in most cases. In the case of the decay rate model, monitoring times of 120 to 150 days were required but the predictions were within about 10% of the ultimate measured BMP in most cases.

Table13 Comparison of prediction methods

	BMP_t/BMP		First-order decay model	
	Monitoring Time (days)	BMP_t/BMP	Monitoring Time (days)	BMP_ultimate /BMP
MSW(C+H)/L>1	30	0.68~0.79	120 (Exclude MSW 8)	0.88~1.03
Green Waste	60	0.49~0.55	150	0.92~1.11
Wood	120	0.60~0.74	120	0.83~1.07
Paper Products	60	0.75~0.91	120	0.96~1.08

4. Conclusions and recommendations

4.1 Conclusions

1) Different inocula result in differences in methane production in the BMP test for 11 MSW samples over a range of (C+H)/L ratios. Sludge and leachate inocula were observed to have higher methane production than the lab-derived culture after about 90 days (median = 26.1% and 20.7%, respectively). The BMP was similar with sludge and leachate inocula.

2) Continuous methane production was observed for all samples after 100 days which indicates that a traditional incubation time of 100 days for BMP test is not sufficient. Different samples showed different methane production trends.MSW, green waste, wood and paper demonstrated a range of total methane yields of 43.0 to 230.0, 53.5 to 75.1, 17.2 to 75.8 and 68.8 to 346.2 mL CH_4/g OS, respectively.

3) A relationship between (C+H)/L ratio and BMP was observed in MSW and green waste samples. However,(C+H)/L ratio was not well correlated with BMP for wood and green waste. The (C+H)/L ratio can be used to approximate the BMP for MSW and green waste samples.

4) An incubation time of 100 days for a BMP test is not sufficient to directly measure the ultimate BMP. However, shorter times (less than 120 days) can be used to estimate the total methane yield. Of the two methods explored, the first order decay model is most reliable for a broad range of samples. The BMP_t/BMP ratio was acceptable for MSW and green waste but should not be generally applied without more data.

5) The recommended incubation time to obtain sufficient data to extrapolate total methane production for MSW ((C+H)/L ratio>1), green waste, wood and paper is 30, 60, 120 and 60 days, respectively, when using BMP_t/BMP method. If using the first order decay method, the recommended monitoring times are 150 days for green waste and 120 days for MSW, wood and paper.

4.2 Recommendations for future research

1) To explore whether the incubation time required to obtain reliable predictions using a first order decay model can be reduced, BMP tests should be repeated with some of the same substrates and a measurement frequency of about 10 days.

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APPENDICES

Appendix A

BMP protocol

1. Inoculum preparation and maintenance

The lab culture is a mixed culture that has been acclimated to grow on dried ground refuse and maintained in lab for over 10 years. This culture has been transferred every two weeks to keep it in an active state. In addition, culture transfer procedure was done two weeks prior to every inoculation of BMP, which sets to minimize background methane production related to inoculum.

1.1 Maintenance medium preparation

Maintenance media is prepared by first adding the following components to a round bottom flask (usually 2L) with a stir bar in the order listed in the Table A-1 Medium for Refuse Consortium Maintenance. Using a 2M NaOH solution to adjust pH to be around 7.2, the solution will appear blue at this point. Then an iron needle is put in to provide 80%/20% mixture of N_2/CO_2 flow to the flask. The flow is kept running until the medium is distributed into each serum bottle. After heating the solution, it needs to be boiled for about 5 minutes. This step can help remove most of the oxygen in the solution. Remove the heat to let the solution cool for 5 minutes under gas flow and add 3.5g/L $NaHCO_3$ powder. Here the medium needs to cool to room temperature while being stirred. During this time, prepare 160-mL serum bottles by weighting approximately 0.4 gram of ground fresh refuse and extract 84 mL of cooled medium with a pipette and put into each bottle under the gas flow. Quickly seal the serum bottles by using black butyl stoppers and aluminum crimps to make them gas-tight. The final step is to autoclave the bottles using a sterilization temperature of 250°C and a sterilization time of 15 minutes. Store the bottles at room temperature.

Table A- 1 Medium for Refuse Consortium Maintenance

Component	**g per liter**
PO_4 solution	100 mL
M3 solution	100 mL
Trace Mineral solution	10 mL
Vitamin solution	10 mL
Yeast extract	0.25 g
Trypticase peptones	0.25 g
Hemin (0.01%)	10 mL
Resazurin (0.1%)	2 mL
Distilled water	758 mL

1.2 Inoculum transfer

First, vent the old culture bottles and de-crimp the bottles and new maintenance media bottles. Add 1 mL of 0.2 M sodium sulfide solution per bottle to reduce the medium. Swirl the bottles to mix in the sulfide. Wait a few minutes for the media to turn faint yellow, which indicates that it has been reduced. 15 mL inoculum from old culture bottles is transferred to each new maintenance media bottle separately, using 60 mL syringe in an anaerobic hood. The newly inoculated bottles are sealed, crimped and stored into the 37°C incubator. Wait for two weeks for the next inoculum transfer and BMP to set up.

2. BMP assays set up

2.1 BMP medium preparation

Firstly, add the following components into a round bottom flask in the order listed in the Table A-1. Notice there is no yeast extract, peptones or hemin this time. The following steps are the same in preparing the maintenance medium before the medium is cooled to room temperature. After cooling, add 10mL of 5% cysteine hydrochloride solution per 1L of solution. The medium will gradually turn pink after continuous stirring. Now dispense 80 mL of the medium using the 100-mL pipette in each of the serum bottles that contain samples or are going to be used as blanks. After that, seal the bottles with stoppers and place the whole set into the 37°C incubator for about two hours until the medium all turn clear. At this point, the medium is now anaerobic and ready to inoculate.

Table A- 2 BMP medium composition

Component	**g per liter**
PO_4 solution	100 mL
M3 solution	100 mL
Trace Mineral solution	10 mL
Vitamin solution	10 mL
Resazurin (0.1%)	2 mL
Distilled water	768 mL

2.2 Inoculation

Vent the 2-week-old inoculum before unstoppering near a venting snorkel using a 23 g needle and a plastic syringe without the plunger. Using 60-mL syringe, dispense 15 mL of inoculum into each serum bottle under s stream of 80%/20% N_2/CO_2. Inoculum should be mixed using a stir bar to make all the 15 mL homogeneous. This step should be fast to avoid oxygen getting into the bottles. After the inoculation, seal and crimp the bottles and incubate at 37°C.

3. Monitoring and manifold flushing procedure

Monitoring frequencies for the samples are every 30 days for the first 180 days and then every 60 days after that. A manifold flushing procedure is conducted after GC analysis by shaking serum bottles and flushing the headspace with 80%/20% N_2/CO_2 to eliminate the CH_4 in both liquid and gas phases. A Quality Check is done after the flushing procedure to ensure the remaining gas phase has a CH_4 concentration less than 400 ppm. Then the set of samples is put back to incubator for next round of incubation.

4. Preparation for basic solutions

4.1 Phosphate Solution

Prepare 1 litercarbonate-free water and dissolve 16.1 g KH_2PO_4 and 31.89 g $Na_2HPO_4{\bullet}7H_2O$. Carbonate-free water is prepared by boiling deionized water and allowing it to cool under O_2-free N_2. Store under N_2 at 4°C.

4.2 M3 Solution

Dissolve 10 g NH_4Cl, 9 g NaCl, 2 g $MgCl_2{\bullet}6H_2O$ and 1 g $CaCl_2{\bullet}2H_2O$ in 1 liter deionized water. Store solution at 4°C.

4.3 Trace Mineral Solution

Table A- 3 Components of Trace Mineral Solution

Component	g per liter
Nitrilotriacetic Acid	1.5 g
$FeSO_4$•$7H_2O$	0.1 g
$MnCl_2$•$4H_2O$	0.1 g
$CoCl_2$•$6H_2O$	0.17 g
$CaCl_2$•$2H_2O$	0.1 g
$ZnCl_2$	0.1 g
$CuCl_2$•$2H_2O$	0.02 g
H_3BO_3	0.01 g
Na MoO_4•$2H_2O$	0.01 g
NaCl	1.0 g
Na_2SeO_3	0.017 g
$NiSO_4$•$6H_2O$	0.026 g
Na_2WO_4•$2H_2O$	0.033 g

Dissolve the nitrilotriacetic acid in 200 mL of hot distilled H_2O and then adjust the pH to 6.5 with KOH. Add this solution to about 600 mL of distilled water and dissolve the components in the order listed. Dilute to one liter. Store in the refrigerator under nitrogen.

Note: Procedure is as described by Kenealy and Zeikus (1981) except for the addition of 0.033 g of Na_2WO_4•$2H_2O$.

4.4 Vitamin Solution

Table A- 4 Components of Vitamin Solution

Component	**g per liter**
Biotin	0.002
Folic Acid	0.002
B_6 (pyridoxine) HCl	0.01
B_1 (thiamine) HCl	0.005
B_2 (riboflavin)	0.005
Nicotinic Acid (niacin)	0.005
Pantothenic Acid	0.005
B_{12} (cyanocobalamin) crystalline	0.0001
PABA (P-aminobenzoic acid)	0.005
Lipoic Acid (thioctic)	0.005
Distilled Water	1000 mL

Add ingredients in the order given and let dissolve. Store in a dark container in the 4°C refrigerator under nitrogen.

4.5 Hemin Solution

Prepare a 0.1% Hemin solution (by weight) and store at 4°C.

4.6 Resazurin Solution

Prepare a 0.01% Resazurin solution (by weight) and store at 4°C.

Appendix B

Additional data analysis

Relationships of production rate drop and prediction results by first-order decay model

$R_{30}/R_t$is defined as the average methane production rate of the first 30 days over the rate of last 30 days used for prediction in the first order decay model. t is the day of last measurement used in the model. Figure B-1 demonstrates R_{30}/R_tratios and prediction results. Very high variations of BMPu/BMP were observed if using first 90 day data and this variation gets smaller when more data points used in this model. Table B-1 lists the values of R_{30}/R_tratios and prediction results. No relevance between R_{30}/R_{120} ratios and performance of the model was observed within a certain type of samples. For example, MSW 9 and MSW 10 had very different R_{30}/R_{120} ratios (19.1 and 7.9 respectively), but the prediction/BMP ratios were close (BMPu/BMP=0.89 and 0.88, respectively.

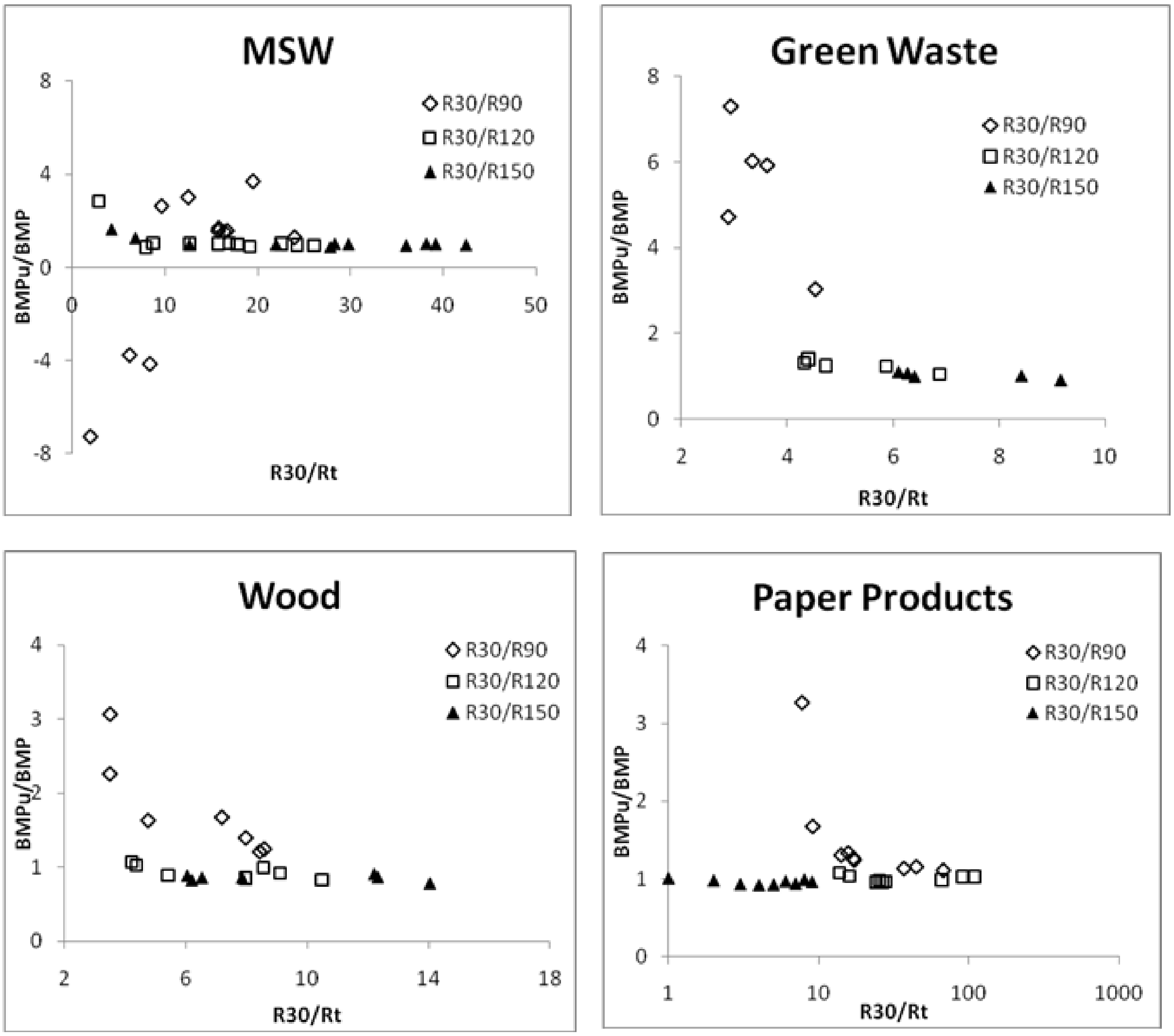

Figure B-1 Relationships of R_{30}/R_t and prediction results.$R_{30}/R_t$is defined as the average methane production rate of the first 30 days over the rate of last 30 days used for prediction in the first order decay model. Paper product doesn’t include the case when $R_{30}/R_t = \infty$.

Table B- 1 Prediction results of first-order decay model

Sample Type	Sample Description	R_{30}/R_{90}[b]	BMPu_90/BMP	R_{30}/R_{120}[b]	BMPu_120/BMP	R_{30}/R_{150}[b]	BMPu_150/BMP
MSW	MSW1	15.7	1.7	16.9	1.0	38.1	1.0
	MSW2	15.7	1.6	15.7	1.0	28.3	1.0
	MSW3	19.4	3.7	22.5	1.0	39.1	1.0
	MSW4	16.6	1.6	17.8	1.0	29.8	1.0
	MSW5	23.9	1.3	24.2	1.0	42.4	1.0
	MSW6	9.6	2.6	12.6	1.0	22.0	1.0
	MSW7	16.3	1.5	26.0	0.9	36.0	0.9
	MSW8	1.9	-7.3	2.8	2.8	4.3	1.6
	MSW9	12.4	3.0	19.1	0.9	27.8	0.9
	MSW10	8.3	-4.2	7.9	0.9	6.8	1.2
	MSW11	6.1	-3.8	8.7	1.0	12.6	1.0
Green Waste	Layer 1	3.3	6.0	4.3	1.3	6.3	1.1
	Layer 2	2.9	7.3	4.4	1.4	6.1	1.1
	Layer 3	3.6	5.9	5.9	1.2	8.4	1.0
	Layer 4	4.5	3.0	6.9	1.0	9.2	0.9
	Layer 5	2.9	4.7	4.7	1.2	6.4	1.0
Wood	HW	3.5	3.1	4.2	1.1	6.0	0.9
	SW	7.2	1.7	8.6	1.0	12.2	0.9
	OSB-HW	8.6	1.2	10.5	0.8	14.0	0.8
	OSB-SW	8.4	1.2	8.0	0.9	7.8	0.9
	PW	8.0	1.4	9.1	0.9	12.3	0.9
	PB	4.7	1.6	5.4	0.9	6.2	0.8
	MDF	3.5	2.3	4.4	1.0	6.5	0.9
Paper Products	OMG 1	44.7	1.2	91.3	1.0	∞[c]	1.0
	OMG 2	7.8	3.3	13.7	1.1	23.9	1.0
	ONP 1	17.2	1.2	28.0	1.0	35.2	0.9
	ONP 2	14.1	1.3	24.1	1.0	29.4	0.9
	ONP 3	17.0	1.3	26.7	1.0	36.0	0.9
	ONP 4	9.1	1.7	16.1	1.0	24.0	1.0
	ONP 5	15.7	1.3	25.4	1.0	35.1	0.9

a. Based on per gram dried sample, instead of organic solids. BMPu is the ultimate BMP predicted in the model.
b. R_{30}/Rt = average methane production rate of the first 30 days/rate of the last 30 days used in model. t is the day of last measurement used in the model. For example, R_{90} is the average methane production rate in days 60 to 90. For MSW samples, the fifth measurements were on day 174 for MSW 1 to 7, 10 to 11 and day 164 for MSW 8 to 9. Therefore R_{150} for MSW were the rate from days 120 to 164/174.
c. R_{150} were nearly zero for OMG1 and OMG2, so R_{30}/R_{150} resulted in infinity.

Appendix C

Original data-see attached spreadsheets

Inocula effect test:

Units: mL CH_4/gram

NCSU ID	Sample Description	Inoculum type and Days		
		Lab-derived Culture Day 93	**Sludge Day 94**	**Leachate Day 89**
03-175A		58.5	79.7	76.8
03-175B		78.4	73.0	84.0
03-175C		74.1	72.8	68.0
Average	**1**	**70.3**	**75.1**	**76.3**
03-201A		117.3	127.9	134.0
03-201B		122.1	132.3	136.7
03-201C		117.9	133.8	140.2
Average	**2**	**119.1**	**131.3**	**137.0**
03-283A		54.3	79.5	75.0
03-283B		64.6	88.5	77.6
03-283C		54.8	77.9	83.0
Average	**3**	**57.9**	**82.0**	**78.5**
04-13A		112.9	116.2	138.0
04-13B		126.0	136.3	140.9
04-13C		123.3	131.3	139.1
Average	**4**	**120.7**	**127.9**	**139.3**
04-15A		127.5	134.3	141.0
04-15B		133.1	134.5	135.1
04-15C		127.3	134.5	141.2
Average	**5**	**129.3**	**134.4**	**139.1**
04-18A		133.5	148.0	153.8
04-18B		130.9	141.8	151.7
04-18C		139.9	152.4	145.3
Average	**6**	**134.7**	**147.4**	**150.3**
05-157A		5.1	8.4	10.1
05-157B		4.8	8.3	7.4
05-157C		4.8	7.8	6.2
Average	**7**	**4.9**	**8.2**	**7.9**
05-159A		11.2	15.2	14.3
05-159B		8.5	15.6	15.4
05-159C		9.8	15.6	12.5
Average	**8**	**9.8**	**15.5**	**14.1**
05-179A		10.8	16.0	15.0
05-179B		11.6	16.2	15.3
05-179C		11.8	17.8	14.8

Average	**9**	**11.4**	**16.7**	**15.0**
05-202A		8.1	13.3	9.4
05-202B		8.0	12.6	12.2
05-202C		9.6	n.m.	10.4
Average	**10**	**8.6**	**13.0**	**10.7**

n.m.=not measured

MSW BMP test:

Units: mL CH_4/gram

NCSU ID	Sample Description	Days													
		0	29	61	93	121	174	213	269	372	427	484	543	608	
03-262 Avg.	MSW 1	0.0	134.4	148.1	157.5	165.2	171.6	173.8	176.2	178.7	179.8	180.7	180.7	182.6	
04-13 Avg.	MSW 2	0.0	102.6	113.5	120.7	127.0	133.7	136.0	138.4	141.4	142.5	n.m.	143.9	144.6	
04-18 Avg.	MSW 3	0.0	120.5	127.9	134.7	139.9	145.5	147.4	149.5	152.3	153.3	n.m.	155.1	155.7	
03-201 Avg.	MSW 4	0.0	102.4	112.3	119.1	124.7	131.0	132.8	134.8	137.6	138.8	139.9	140.9	142.0	
04-15 Avg.	MSW 5	0.0	115.4	124.0	129.3	133.9	138.9	141.1	143.5	146.8	148.4	n.m.	150.6	151.7	
03-175 Avg.	MSW 6	0.0	55.7	63.9	70.3	74.6	79.2	81.1	83.2	86.2	87.3	88.5	89.6	90.7	
03-283 Avg.	MSW 7	0.0	49.5	54.6	57.9	59.7	62.3	63.0	64.5	66.7	67.6	68.4	69.1	69.9	
05-157 Avg.	MSW 10	0.0	3.9	4.4	4.9	5.4	6.4	7.1	7.7	8.4	8.6	n.m.	9.0	9.2	
05-159 Avg.	MSW 11	0.0	7.3	8.5	9.8	10.7	11.7	12.3	13.3	14.3	14.7	n.m.	15.3	15.5	
NCSU ID	Sample Description	Days													
		0	32	61	93	120	164	195	240	310	373	425	484	540	611
05-202 Avg.	MSW 8	0.0	4.5	6.2	8.6	9.9	11.3	12.0	12.7	13.4	13.9	14.3	14.5	14.8	15.2
05-179 Avg.	MSW 9	0.0	9.8	10.6	11.4	11.9	12.3	12.8	13.3	13.9	14.4	14.7	14.8	15.1	15.3

n.m= not measured

Green Waste BMP test:

Units: mL CH_4/gram

NCSU ID	Sample Description	Days												
		0	30	61	91	119	150	196	256	363	423	489	555	606
09-543 Avg.	Layer 1	0.0	7.0	9.5	11.6	13.1	14.3	15.6	16.7	17.3	18.0	18.6	19.1	19.5
09-544 Avg.	Layer 2	0.0	8.0	11.3	14.1	15.7	17.1	18.6	19.8	20.4	21.1	21.7	22.3	22.6
09-545 Avg.	Layer 3	0.0	7.5	10.1	12.2	13.4	14.3	15.3	16.2	16.7	17.3	17.8	18.3	18.6
09-546 Avg.	Layer 4	0.0	10.4	13.7	16.0	17.4	18.5	19.9	21.3	22.0	22.9	23.7	24.5	24.9
09-547 Avg.	Layer 5	0.0	8.8	13.4	16.4	18.2	19.6	21.3	22.9	23.8	24.9	25.8	26.6	27.1

Wood samples BMP test:

Units: mL CH_4/gram

NCSU ID	Sample Description	Days														
		0	29	63	90	119	150	212	267	329	372	426	485	547	601	682
07-151 Avg.	HW	0.0	16.7	37.7	42.2	46.1	49.1	52.4	54.5	56.7	58.4	61.3	63.8	66.5	70.0	75.8
07-150 Avg.	SW	0.0	10.6	12.9	14.3	15.5	16.4	17.7	18.5	19.2	19.7	20.1	20.5	20.9	21.2	21.6
08-1876 avg	OSB-SW	0.0	7.4	9.2	10.0	11.0	12.0	13.2	14.0	14.7	15.2	15.7	16.1	16.5	16.8	17.2
07-152 Avg.	OSB-HW	0.0	34.5	45.2	48.9	52.2	54.8	58.1	60.4	62.6	64.5	70.4	73.3	75.4	77.1	79.5
07-153 Avg.	PW	0.0	8.7	10.9	11.9	12.9	13.6	14.7	15.5	16.1	16.5	16.9	17.3	17.6	17.9	18.3
07-154 Avg.	PB	0.0	8.1	11.4	12.9	14.4	15.8	17.7	19.2	20.6	21.5	22.4	23.3	24.2	25.0	26.1
07-155 Avg.	MDF	0.0	6.5	10.3	12.1	13.6	14.7	16.5	17.8	18.9	19.5	20.8	21.7	22.5	23.2	24.2

Paper Products BMP test:

Units: mL CH_4/gram

NCSU ID	Sample Description	Days														
		0	32	60	89	120	150	180	227	287	364	440	484	542	568	634
08-78 Avg.	ONP 1	0.0	51.3	57.7	60.4	62.1	63.5	64.5	65.7	66.8	67.9	68.6	68.9	69.3	69.5	70.0
08-79 Avg.	ONP 2	0.0	38.2	43.7	46.1	47.7	48.9	49.6	50.6	51.7	52.9	53.6	53.9	54.4	54.6	55.1
		Days														
NCSU ID	Sample Description	0	29	61	91	123	149	185	223	268	369	427	485	556	611	
08-80 Avg.	OFF 1	0.0	217.4	231.9	235.3	237.5	237.5									
08-852 Avg.	OFF 2	0.0	220.2	242.9	249.1	252.7	254.7	257.0	258.4	259.8	263.0	264.2	265.3	266.6	267.6	
		Days														
NCSU ID	Sample Description	0	30	61	91	119	150	196	256	317	363	423	489	555	606	
08-81 Avg.	OMG 1	0.0	156.3	165.3	168.8	170.4	170.4	170.4								
08-82 Avg.	OMG 2	0.0	36.9	42.8	47.5	50.1	51.7	53.2	54.4	55.3	55.9	56.7	57.3	57.8	58.1	
08-1985 Avg.	ONP 3	0.0	48.7	55.4	58.3	60.0	61.4	63.1	64.2	65.4	66.0	66.8	67.5	68.1	68.6	
08-1986 Avg.	ONP4	0.0	48.4	61.6	66.9	69.8	71.8	73.8	75.4	76.5	77.2	78.1	78.9	79.7	80.3	
08-1987 Avg.	ONP 5	0.0	47.4	53.9	57.0	58.7	60.1	61.5	62.5	63.4	64.0	64.7	65.3	65.9	66.3	