



Contents lists available at ScienceDirect

## Waste Management

journal homepage: [www.elsevier.com/locate/wasman](http://www.elsevier.com/locate/wasman)

# Effects of temperature and particle size on the biochemical methane potential of municipal solid waste components

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## ARTICLE INFO

## Article history:

Received 19 July 2017

Revised 31 October 2017

Accepted 5 November 2017

Available online xxx

## Keywords:

Biochemical methane potential

Anaerobic digestion

Municipal waste

Landfill

Biodegradation

## ABSTRACT

The effects of temperature and substrate size on the biochemical methane potential (BMP) assay were tested using eight municipal solid waste components. Two sample sizes were tested; size-reduced particles ( $x < 2$  mm) which are typically used for BMP assays and unground samples ( $x > 20$ – $100$  mm) more similar to an as-disposed condition. Two incubation temperatures (35 and 55 °C) were tested for each component. BMPs for office paper, newspaper, paperboard, and coated paper displayed little difference with regards to temperature or particle size. Mesophilic corrugated cardboard BMPs were significantly greater than their thermophilic counterparts. Hardwood, softwood, and cotton BMPs varied with particle size and temperature. Particle size reduction may increase the bioavailable carbon compounds for wood, but this step was not necessary to achieve similar methane yields for paper products. Extrapolating BMP results to predict landfill methane generation may have greater uncertainty for wood wastes and cotton textiles than paper products.

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## 1. Introduction

The biochemical methane potential (BMP) assay has become a standard tool for solid waste engineers to predict methane generation from municipal solid waste (MSW) components (Elbeshbishy et al., 2012; Owens and Chynoweth, 1993; Wang and Barlaz, 2016). Developed by Owen et al. (1979), the BMP assay quantifies the maximum amount of methane that can be generated from a substrate experimentally. The method requires a small mass of sample relative to the anaerobic inoculum, ensuring complete degradation that is limited only by carbon bioavailability in the substrate. The first step in a BMP experiment is preparation of the sample for analysis, generally by particle size reduction (Owens and Chynoweth, 1993). Size reduction is suggested for MSW to create a more uniform sample; a substrate that is naturally heterogeneous and challenging to study with smaller masses (Hansen et al., 2004). This step is considered to be at the discretion of the researcher and the objective of the research, but it is encouraged especially for smaller assays (100–250 mL bottle). Both substrate particle size and inoculation temperature are understood to effect degradation during the 60-day incubation period (Angelidaki et al., 2009).

Reducing particle size has been shown to speed the rate of methane generation by increasing available surface area and

thereby minimizing the rate-limiting hydrolysis step in digestion (Vavilin et al., 2008). Palmowski and Müller (2000) found reducing particle size of food wastes did not increase the BMP, but lignocellulosic BMPs were positively impacted by size-reduction. Because lignin is found in many paper types, it may be that size-reduction liberates previously-unavailable cellulose and hemicellulose for methanogenesis, increasing overall methane yield. In an experiment to identify methane yields of MSW components, Eleazer et al. (1997) did not size-reduce coated paper, known to contain inorganic clay, specifically for this reason. Pommier et al. (2010) performed BMPs on mixed paper/cardboard waste, reporting no improvement of BMP at reduced particle size. However, categorizing one sample type as “mixed” leads to uncertainty if the material was identical in both conditions, which could significantly impact the results.

While landfilling remains the primary means of waste management worldwide, the biodegradable components of MSW are increasingly considered as a viable substrate for anaerobic digestion (Jokela et al., 2005). Digesters are operated at mesophilic (~35 °C) or thermophilic (~55 °C) conditions. Thermophilic reactors may be preferred because of the reduced retention times/increased digestion rates, though the higher temperatures for operation often come at a higher cost. Kinetics of anaerobic digestion indicate that as temperature increases in a biologically-viable range, the rate of degradation will also increase (Veeken and Hamelers, 1999). Cumulative methane production is not believed

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to be impacted by temperature; however, the anaerobic consortia in mesophilic and thermophilic conditions are not identical (Karakashev et al., 2005; Sekiguchi et al., 1998) and the inoculum must be appropriate for the incubation conditions (Angelidaki et al., 2006). Previous experiments showing higher methane yields at thermophilic conditions may have been a result of terminating reactions before ultimate mesophilic yields were reached (Harmon et al., 1993).

Methanogenesis, the catabolic process responsible for methane formation in landfills, is a biochemical process that requires a diverse microbial community to effectively break down larger chemical structures to those that can be consumed by methanogens (Barlaz et al., 1990; Vavilin et al., 2008). The Intergovernmental Panel on Climate Change (IPCC) originally published a methodology to predict methane generation from landfills that indicated the fraction of anaerobically degradable organic carbon (DOC<sub>f</sub>) was a function of temperature, based on a kinetic model (IPCC, 1997). This methodology has since been replaced with a non-kinetic assumption (IPCC, 2006). Based on the limited available literature, it is uncertain if temperature impacts the methane yield of lignocellulosic MSW components.

While research clearly indicates smaller particle sizes and higher temperatures will increase the rate of methane production, the same cannot be said for the cumulative yield (Raposo et al., 2012). Ultimately, it is not clear if particle size and/or temperature impact the BMP of lignocellulosic MSW components. As the BMP is used for a range of purposes (e.g., digester performance, life-cycle assessments, greenhouse gas inventories), it is important to understand how the experimental parameters impact results and to determine if those parameters are acceptable for extrapolating or applying BMP data to other scenarios. In this research, samples of office paper, newspaper, coated paper, corrugated cardboard, paperboard, cotton, dimensional hardwood lumber (oak), and softwood lumber (cedar) were separately analyzed by BMP at two different particle sizes ( $x < 2$  mm and  $x > 20$ –100 mm) and two temperatures (35 °C and 55 °C) to determine if these two parameters impact the measured BMP of each MSW component.

## 2. Methods and materials

### 2.1. Physical analyses

Office paper, newspaper, corrugated cardboard, paperboard, and coated paper were collected prior to disposal on the University of Florida (UF) campus. Office paper was unused, blank printing paper. Newspaper was collected from a newspaper stall on the UF campus. Cardboard was collected from a recycling bin prior to disposal. Glossy coated paper was taken from inner pages of a printed magazine. Paperboard was a non-corrugated beverage package collected prior to disposal. Hardwood was an oak lumber, softwood was a cedar lumber, and cotton was an unused cotton cloth.

For each MSW component, size-reduced and larger, as-disposed samples were analyzed by BMP. Where possible, the same parent material was used in all physical and biochemical analyses. For example, the dimensional hardwood sample was purchased from a local retailer and fractions were cut from the sample for each protocol: moisture content, volatile solids, and BMP. In other cases, such as office paper, where the mass of a single piece of paper was not sufficient for all analyses, material was taken from the same ream of paper.

Samples were weighed as-received to determine a wet weight. Moisture content (MC) was quantified as the mass loss after oven heating at 105 °C for 24 h. Volatile solids (VS) content was measured as the mass loss from the total solids after 4 h at 550 °C in a muffle furnace.

### 2.2. Biochemical methane potential

The BMP assay was used to evaluate methane yields of municipal solid waste components at two different temperatures (35 and 55 °C) and particle sizes ( $x < 2$  mm and  $x > 20$ –100 mm). The method uses a dense nutrient broth, anaerobic inoculum, and an oxidizing indicator to ensure anaerobic conditions throughout the incubation (Owen et al., 1979). Mesophilic sludge was collected from a digester maintained in the Solid and Hazardous Waste Laboratory at UF. Thermophilic sewage sludge was collected from a wastewater treatment plant anaerobic digester in Lakeland, FL. Sludge was pre-incubated without substrate for approximately 5 days to reduce background activity, in line with the method by Angelidaki et al. (2009). BMP vessels were purged with high purity nitrogen for 3 min each to remove oxygen from the reactor headspace and incubated at  $35 \pm 1$  or  $55 \pm 2$  °C. Positive (cellulose) and blank (no substrate, inoculum only) controls were used to evaluate inoculum performance.

As-disposed particles were created by cutting or milling a large particle (smallest side was 20–100 mm) from the original material. Another portion was removed from the original material and size-reduced with a commercial blender (Blendtec) to pass a 2 mm sieve, creating significantly greater surface area for size-reduced samples. Waste components were inoculated at a substrate-to-inoculum ratio (S/I) of 0.02 g VS/mL; equivalent to a liquid-to-solid ratio of 5000:1 mL/g VS (ASTM International, 1992). Size-reduced samples were incubated in 250 mL serum bottles with 100 mL of anaerobic growth media/inoculum. To allow for a sufficiently large particle size to be analyzed, the sample size and reactor size was multiplied by 10 (i.e., 2 g VS loaded into a 2000 mL media bottle with 1000 mL inoculum). Fig. 1 shows (A) office paper and (B) hardwood prior to bottling with size-reduced and as-disposed samples.

Assays were performed in triplicate for both particle sizes and temperatures. Biogas quantity was measured with gastight volumetric syringes (Cadence Science) held horizontally (Owen et al., 1979). Biogas composition was analyzed in a gas chromatograph equipped with a thermal conductivity detector using a 1 m ResTek ShinCarbon ST packed column (Shimadzu GC-8A). Injector and detector temperatures were set to 110 and 100 °C, respectively. Methane generation was normalized to standard temperature and pressure (STP; 0 °C and 760 mm Hg) by accounting for ambient pressure, water vapor pressure, and incubation temperature. Water vapor was accounted for by subtracting the water vapor pressure at the incubation temperature from the total biogas generation, as shown in Eq. (1). Samples were measured immediately after removal from the incubators to keep internal temperatures and pressures consistent throughout the experiment

$$V_{CH_4,STP} = V_{CH_4} \times \left( \frac{273.15}{273.15 + T_{inc}} \right) \times \left( \frac{P_a - P_{w,inc}}{P_{STP}} \right) \quad (1)$$

where  $V_{CH_4,STP}$  is the volumetric methane generation at STP;  $V_{CH_4}$  is the volume of methane in the biogas,  $T_{inc}$  is the incubation temperature recorded before opening the incubator,  $P_a$  is the ambient pressure of the laboratory;  $P_{w,inc}$  is the water vapor pressure at  $T_{inc}$ ; and  $P_{STP}$  is the pressure at STP (760 mm Hg). This equation was used for each methane measurement and the cumulative values are reported in the following sections. Methane production from the blank controls were subtracted from samples to evaluate methane generation from only the substrate. To identify if a statistical difference between any of the two BMP sets existed, a *t*-test was applied to the final BMP values (Kebbekus and Mitra, 1997). Statistical formulas and final BMP data are given in Appendix A of the Supplemental Information (SI). The results of the statistical analysis are reported in the following section.

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