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Influence of headspace pressure on methane production in Biochemical Methane Potential (BMP) tests

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ABSTRACT

The biochemical methane potential test is the most commonly applied method to determine methane production from organic wastes. One of the parameters measured is the volume of biogas produced which can be determined manometrically by keeping the volume constant and measuring increases in pressure. In the present study, the effect of pressure accumulation in the headspace of the reactors has been studied. Triplicate batch trials employing cocoa shell, waste coffee grounds and dairy manure as substrates have been performed under two headspace pressure conditions. The results obtained in the study showed that headspace overpressures higher than 600 mbar affected methane production for waste coffee grounds. On the contrary, headspace overpressures within a range of 600–1000 mbar did not affect methane production for cocoa shell and dairy manure. With the analyses performed in the present work it has not been possible to determine the reasons for the lower methane yield value obtained for the waste coffee grounds under high headspace pressures.

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1. Introduction

Anaerobic digestion (AD) is an attractive sustainable environmental technological process that stabilizes organic wastes and produces renewable energy in the form of biogas and nutrient rich and hygienized digestate (Nkemka et al., 2015). Organic matter can be characterized by either chemical or biological methods. Different methods have been recommended to determine the anaerobic biodegradability and methane potentials for organic substrates (Lesteur et al., 2010). The Biochemical Methane Potential (BMP) test is the most commonly applied method to determine biogas and methane production from organic substrates (Angelidaki et al., 2009; Godin et al., 2015; Koch et al., 2015; Strömberg et al., 2014). The method is performed at laboratory scale and provides information on both the quantity of methane which can be produced from a specific substrate, and also the speed at which the production occurs.

Despite the wide use of BMP tests, no commonly accepted experimental procedure yet exists that is based on a standardized protocol for the execution of the test (Koch et al., 2015; Murovec et al., 2015). Koch et al. (2015) reported that there are two main protocols applied for performing the BMP test. Of these, one is the method published by the Task Group for the Anaerobic Biodegradation, Activity and Inhibition of the Anaerobic Digestion Specialist Group of the International Water Association (IWA) in 2009 (Angelidaki et al., 2009). The other one is the technical guideline VDI 4630 (VDI 4630, 2006), performed by the Association of German Engineers (Verein Deutscher Ingenieure, VDI). The ISO Guideline 11734 (ISO 11734, 1995) also describes the method to evaluate the ultimate anaerobic biodegradability of organic compounds in digested sludge.

At laboratory scale there are basically two methods to measure biogas production in BMP tests: volumetrically by providing constant pressure and measuring the volume of biogas by displacement volume devices, or manometrically by keeping the volume constant and measuring increases in pressure (Rozzi and Remigi, 2004; Parajuli, 2011). The first time that portable pressure transducer devices were used in methanogenic activity tests date from 1988 (Concannon et al., 1988). The use of pressure transducers simplifies the set-up of the experiment. The volume of gas

Abbreviations: AD, anaerobic digestion; BA, bicarbonate alkalinity; BMP, Biochemical Methane Potential; COD, chemical oxygen demand; $COD_{biodegradable}$, biodegradable COD; COD_{VFA}, COD due to volatile fatty acids; CS, cocoa shell; DM, dairy manure; P_{amb}, ambient pressure; $P_{headspace}$, headspace overpressure; P_{STP}, standard pressure; T_{STP}, standard temperature; TKN, Total Kjeldahl Nitrogen; TS, Total Solids; $V_{headspace}$, headspace volume; VFA, volatile fatty acids; VS, Volatile Solids; WCG, waste coffee grounds.

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produced can be calculated from the measured overpressure. A gas release device must be available to reduce pressure to avoid high pressures during the test. Headspace pressure is an experimental condition that can affect the test (Angelidaki et al., 2009). Recently, Koch et al. (2015) studied the influence of the headspace flushing gas on methane production in the BMP test. Although some studies have been performed on the effect of high pressure on the performance of the continuous AD process (Chen et al., 2014; Lindeboom et al., 2011, 2012), to the best of our knowledge there are no previous works in which the effect of pressure accumulation in the headspace reactors in BMP tests had been studied. According to these studies, CO_2 and pH in the medium, as well as bacteria growth, are affected by high pressures.

The importance of headspace pressure accumulation lies in the fact that the BMP test generally takes 30–60 days (Labatut et al., 2011). When venting is done manually, it might not be possible to measure the pressure and release the produced gas daily during the experiment. In addition, it might not be necessary to take very frequent pressure measures and subsequent venting which would also reduce the time and resources (chromatography for methane determination) used in the test.

This study is a first approach to evaluate the impact of headspace pressure accumulation on the performance of BMP tests by manometric biogas volume determination. The impact of this experimental parameter on the calculated BMP has been investigated for three complex organic wastes: dairy manure, waste coffee grounds and cocoa shell.

2. Materials and methods

2.1. Substrates and inoculum

Dairy manure (DM) was collected from the cow house of a 500-free stall dairy cow farm in the Santander area (Northern coast of Spain). Waste coffee grounds (WCG) were collected from the cafeteria of the Civil Engineering Faculty in the University of Cantabria. Cocoa shell (CS) was obtained from a dairy milk processor that manufactures milk chocolate products. A coffee mill was used to reduce the CS particle size (size <1 mm). Anaerobically digested liquid fraction of dairy manure was used as inoculum (I). Previous to the BMP tests, the inoculum was degassed for five days at 38 °C.

2.2. Experimental set-up

Because the objective of the work was to study the effect of headspace pressure accumulation on methane production, the BMP tests did not follow any of the standardized protocols above mentioned.

Two experimental conditions were compared. For the first condition (P0), the reactors were initially vented daily as was necessary to maintain low pressures in the headspace. After that, the reactors were vented nearly every day. In this way, high continuous overpressures were avoided. For the second condition (P1), the reactors were vented when the headspace overpressure reached a threshold of 800 ± 200 mbar. Three control reactors (one for each substrate) were used to follow the pressure evolution of P1 reactors. To compare the results from experiments P0 and P1, a statistical analysis was performed to determine if the differences between both the experiments were significant or not for the different substrates tested. The statistical analysis was performed using SPSS software. The significance of the differences between values obtained under different pressure conditions was assessed using the Student's *t*-test. Values of $P < 0.05$ were considered significant.

Table 1
Characteristics of cocoa shell, waste coffee grounds, dairy manure and inoculum used in the present study (Mean ± SD, $n = 3$).

Parameter	CS	WCG	DM	I
TS (%)	89.9 ± 1.1	40.6 ± 0.3	13.6 ± 0.4	2.26 ± 0.04
VS (%)	82.3 ± 1.2	40.0 ± 0.3	11.9 ± 0.4	1.35 ± 0.03
VS/TS	0.92 ± 0.01	0.99 ± 0.01	0.88 ± 0.01	0.60 ± 0.01
TKN (g N kg^{-1} TS)	25.3 ± 0.2	23.9 ± 0.3	23.0 ± 0.9	44.5 ± 1.2
C/N[a]	20.2 ± 0.1	22.9 ± 0.1	21.2 ± 0.4	7.5 ± 0.1
pH	–	–	–	7.7 ± 0.0
BA (g $CaCO_3$ L^{-1})	–	–	–	18.9 ± 0.1

[a] C/N was calculated assuming the carbon content of substrates are 55% of the VS content (Adams et al., 1951).

Table 2
Data related to the BMP tests of the studied samples under P0[a] and P1[a] conditions. Amounts of substrate, water, inoculum and VS_I/VS_S ratios. Mean values ± SD from triplicate assays.

	Substrate (g)	Inoculum (g)	Water (g)	VS_I/VS_S
CS	0.66 ± 0.01	74.34 ± 0.01	100 ± 0.01	1.85 ± 0.03
WCG	1.65 ± 0.02	73.35 ± 0.02	100 ± 0.01	1.50 ± 0.02
DM	4.66 ± 0.11	70.34 ± 0.11	100 ± 0.01	1.87 ± 0.05

[a] P0 corresponds to frequent venting (low headspace overpressure conditions); P1 condition corresponds to headspace overpressure within the range 800 ± 200 mbar.

For each substrate and condition, the tests were carried out in 250-mL triplicate serum bottles capped with rubber septum sleeve stoppers. Aiming to keep the pressures within the selected range for P1 conditions, and based on earlier assays, each reactor was filled with 175 g of a mixture consisting of dechlorinated tap water, inoculum (I) and substrate (S) (see Table 2). Nitrogen was flushed to remove the air in the headspace of the bottles. Thereafter, all the reactors were placed in an incubator at 38 °C for a period of 35 days. All the reactors were manually agitated once a day. Three blanks with 100 g of water and 75 g of inoculum were also tested to measure methane potential of the inoculum. Results are expressed as means ± SD subtracting methane production from the inoculum.

2.3. Numerical calculations

The cumulated volumes of methane were calculated by the cumulative summation of methane volumes determined each time headspace pressure was measured and the biogas was released by venting. Eq. (1) shows the conversion of headspace pressure to volume of biogas at standard pressure and temperature based on the ideal gas law.

$$V_{biogas\_STP} = V_{headspace} \cdot \frac{P_{headspace}}{P_{STP}} \cdot \frac{T_{STP}}{T} \quad (1)$$

In Eq. (1), V_{biogas_STP} is the volume of biogas, adjusted to standard pressure and temperature (0 °C, 1 atm), and produced between two venting operations. $V_{headspace}$ is the reactor headspace volume, $P_{headspace}$ is the manometric pressure measured in the headspace, P_{STP} is the standard pressure (1013.25 mbar), T_{STP} is the standard temperature in K (273.15 K) and T is the operation temperature (311.15 K). In Eq. (2) the conversion of the methane biogas content from wet to dry conditions is shown.

$$\%CH_{4\_dry} = \%CH_{4\_wet} \cdot \left(1 - \frac{P_{vap}}{P_{amb} + P_{headspace}}\right) \quad (2)$$

In Eq. (2), $\%CH_{4\_dry}$ is the biogas methane content in dry gas conditions whereas $\%CH_{4\_wet}$ is the analyzed biogas methane content (wet conditions). P_{vap} is the vapor pressure of water at

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