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Towards eliminating systematic errors caused by the experimental conditions in Biochemical Methane Potential (BMP) tests

Sten Strömberg [a,*], Mihaela Nistor [b], Jing Liu [a,b]

[a] Department of Biotechnology, Lund University, Getingevägen 60, 221 00 Lund, Sweden
[b] Bioprocess Control, Scheelevägen 22, 223 63 Lund, Sweden

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ABSTRACT

The Biochemical Methane Potential (BMP) test is increasingly recognised as a tool for selecting and pricing biomass material for production of biogas. However, the results for the same substrate often differ between laboratories and much work to standardise such tests is still needed. In the current study, the effects from four environmental factors (i.e. ambient temperature and pressure, water vapour content and initial gas composition of the reactor headspace) on the degradation kinetics and the determined methane potential were evaluated with a 2^4 full factorial design. Four substrates, with different biodegradation profiles, were investigated and the ambient temperature was found to be the most significant contributor to errors in the methane potential. Concerning the kinetics of the process, the environmental factors' impact on the calculated rate constants was negligible. The impact of the environmental factors on the kinetic parameters and methane potential from performing a BMP test at different geographical locations around the world was simulated by adjusting the data according to the ambient temperature and pressure of some chosen model sites. The largest effect on the methane potential was registered from tests performed at high altitudes due to a low ambient pressure. The results from this study illustrate the importance of considering the environmental factors' influence on volumetric gas measurement in BMP tests. This is essential to achieve trustworthy and standardised results that can be used by researchers and end users from all over the world.

1. Introduction

As anaerobic digestion of organic material is becoming more and more established as a sustainable approach for waste management and energy production, the demand and necessity to find suitable feedstock is continuously increasing. A method to explore and determine the feasibility of a material to serve as a substrate in anaerobic digestion is the Biochemical Methane Potential (BMP) test. This assay provides information on how much and how fast the material can be degraded under optimal batch conditions, which are valuable parameters in the design and operation of a biogas plant (Koch and Drewes, 2014; Lesteur et al., 2010; Moody et al., 2009). It is also a good tool to identify and develop new indicators for the evaluation of potential feedstock sources (Buffiere et al., 2006). Furthermore, the correlation between data from BMP test and full scale operation has been investigated and successfully demonstrated with regards to the material's degradability (Batstone et al., 2009; Jensen et al., 2011).

As the biogas sector grows, the demand for energy rich feedstock increases. Ultimately, this development will lead to a market with suppliers of energy rich waste products or other feedstock on one side, and consumers, in the form of biogas producers that are willing to pay for their raw material, on the other side. In order to price the material properly, it will be in the buyer's interest to evaluate the quality and biomethane producing potential of the feedstock. This will be especially important for waste streams where the quality can vary substantially and, for this particular purpose, BMP tests could be an excellent analysis tool (Mahanty et al., 2014). However, in order to get a broad acceptance, the test results need to be reliable and standardised.

Nowadays there are many international and national standards available on how to perform a BMP test, but they differ in the experiment set-up and are many times modified and adapted to the specific researcher's purpose (Otero et al., 2011; Raposo et al., 2011a). Due to this, it is often difficult to evaluate results from different studies as the values and level of information can differ substantially. Today, there is general agreement that there

* Corresponding author. Tel.: +46 46 222 81 93; fax: +46 46 222 47 13.
E-mail addresses: sten.stromberg@biotek.lu.se (S. Strömberg), mn@bioprocesscontrol.com (M. Nistor), jing.liu@biotek.lu.se (J. Liu).

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is need for a standardised and general procedure on how to perform an analysis of the anaerobic biodegradability and biomethane potential of a substrate (Angelidaki et al., 2009; Wulf et al., 2011). This issue has been addressed by an international task group, specialised on the harmonisation of anaerobic biodegradation determination, such as activity and inhibition assays (ABAI-TG), which recently published instructions on what to consider when performing such a test (Angelidaki et al., 2009). Nevertheless, although the instructions have been followed in detail, deviations in the results among different laboratories have been observed; a three round inter-laboratory study in Germany, following standard VDI 4630, generated a variation coefficient of 8% in the reported values for the specific methane yield of cellulose (Wulf et al., 2011). Another international inter-laboratory study involving 19 different labs (Raposo et al., 2011a), reported variations of 15–37% for the standard samples cellulose, starch and gelatine as well as a more conventional substrate in the form of mung bean. Removing statistical outliers reduced the relative standard deviation to 8–11%. It should be noted that this study did not provide any detailed instructions on how to set up the test, instead the participants were instructed to report the conditions in detail to the organisers. A common aspect for both these studies was that no uniform equipment set-up was used to quantify the gas production and, even if a strict protocol was followed for the German study, several of the tasks required human effort which always can lead to deviations. Therefore, the use of an automated laboratory system that is specifically designed for BMP tests should reduce the relative deviation by minimising the human errors. In fact, one issue that is not fully addressed in most standard procedures is the type of equipment and applied experiment set-up, which many times are self-developed and specific for each laboratory.

Besides the level of biodegradability of the material, the results from a BMP test also provide valuable information about the rate of the degradation process that, together with the BMP value, offers key information in the selection of the most suitable type of substrate for the process. Furthermore, it also may provide valuable knowledge in the choice of organic loading rate and retention time of a full-scale plant (Chynoweth et al., 1993; Lesteur et al., 2010). However, this aspect should be regarded with some caution as the dynamic conditions differ in a continuously operated digester compared to a batch one. Batstone et al. (2009), Jacobi et al. (2012) and Jensen et al. (2011) found the rate constants in a continuous process to be larger compared to what was achieved in the BMP tests, whereas Souza et al. (2013) experienced a slight overestimation in the methane production when using kinetic constants calibrated from BMP test data. Therefore, the use of kinetic parameters determined through batch testing should be considered only as a first approximation for its application in predicting the behaviour of a continuous digester (Donoso-Bravo et al., 2011).

The kinetics of the degradation is arguably even more sensitive to the experimental conditions compared to the methane yield, as it is highly dependent on the microbial activity and sample accessibility (Jensen et al., 2011). Another factor that may influence the values of the kinetic constants is how the data is sampled and what type of cost function is used to calculate them. Data may be collected at fixed time intervals or volumes or, alternatively, manually at irregular intervals of these. All of which will have significant impact on the calculated parameters. An example of the complex nature and difficulties in standardising the kinetic constants is offered by the inter-laboratory study performed by Raposo et al. (2011a) which obtained relative standard deviations between 55% and 68% for the reported first order rate constants of the studied samples.

A common problem when comparing results from different sources is the various ways of presenting the values for the quantitative gas measurements (Walker et al., 2009; Wulf et al., 2011). Corrections to standard conditions with regard to temperature and pressure are often poorly described and/or presented using different reference values, which could lead to discrepancies of up to 10% in the corrected volume (Walker et al., 2009). Another factor that is not addressed in many corrections is the water content in the gas. At normal ambient temperatures (i.e. 10–40 °C), approximately 1–7% of the gas volume is water vapour which should be subtracted from the reported values (VDI 4630, 2006). Furthermore, it is also important to monitor the instantaneous temperature and pressure for each measurement in order to calculate the produced gas volumes correctly. However, in practice, a fixed room temperature and pressure is often assumed, although these values can vary substantially. A study investigating the room temperature in 24 different laboratories reported values between 16 and 27 °C with an average of 21.5 °C (Davison and Chiba, 2003). Furthermore, pressure variations between 1000 and 1060 mbar were registered during a 34-day long BMP test performed in Lund, Sweden (Strömberg et al., 2012).

This study aims to evaluate the individual and combined effect coming from neglecting temperature, pressure, water vapour and initial headspace gas composition on quantitative gas measurements in BMP tests. The impact of these experimental parameters on accumulated volume, calculated BMP value and kinetic rate constants are investigated for four sample types and further analysed at some extreme temperature and pressure conditions registered at different geographical locations around the world.

2. Background

2.1. Biochemical Methane Potential (BMP) test

The Biochemical Methane Potential test is an assay that is used to determine the biodegradability and potential to produce methane under anaerobic conditions for organic materials. The investigated material is mixed with an anaerobic bacteria culture, normally sampled from an active biogas plant, and incubated for a period of 30–60 days (Labatut et al., 2011). For optimal performance, the mixture should be kept at a stable temperature, normally at about 37 or 52 °C, and continuously mixed to minimise mass transfer limitations. The organic material, often called substrate, is degraded through a multistep biochemical process with the gaseous compounds methane and carbon dioxide as the major final products. Since only the amount of methane is of interest, the carbon dioxide is often removed using a scrubber agent (e.g. ethylamines, alkaline solution) or, alternatively, the gas composition is measured regularly to compensate for the content of other gases. However, at least for volumetric methods based on water displacement, it is preferred to remove the carbon dioxide physically since some parts of it will always dissolve in the liquid phase leading to inaccurate measurements (Rozzi and Remigi, 2004; Walker et al., 2009). The quantity of the produced gas is most commonly determined using methods based on either manometric or volumetric principles (Raposo et al., 2011b).

The BMP value is presented as the volume of methane per gram of organic material, often defined by volatile solids (VS), chemical oxygen demand (COD) or biological oxygen demand (BOD). For standardisation purposes the BMP value should be presented using the same type of unit and, as most BMP tests today are performed with rather solid material, VS should be regarded as the most suitable choice. However, as many wastewater types might be too liquid to allow reliable VS measurements, characterisation based on COD might be necessary in certain cases. In order to avoid inhibition due to accumulation of intermediate products it is important to have an optimum ratio between inoculum and substrate. It is therefore recommended to have at least two times more inoculum

compared to substrate based on VS amount (Cabbai et al., 2013). Since the bacterial inoculum also contains biodegradable material, the gas originating from this should be considered. Therefore, a sample containing only inoculum, often referred to as blank, is generally tested in parallel with the investigated sample (Hansen et al., 2004). Alternatively, the inoculum can be degassed in order to minimise its gas production during the test. If a blank sample is used, the BMP of the investigated substrate can be calculated according to Eq. (1).

$$\mathrm{BMP} = \frac{V_S - V_B \frac{m_{I,S}}{m_{I,B}}}{m_{S,S}} \quad (1)$$

In Eq. (1), V_S is the accumulated volume of gas coming from the substrate sample (substrate and inoculum), V_B is the volume coming from the blank sample (inoculum), $m_{I,S}$ is the organic material amount of inoculum in the substrate bottle, $m_{I,B}$ is the organic material amount of inoculum in the blank bottle and $m_{S,S}$ is the organic material amount of substrate in the substrate bottle.

Since anaerobic digestion is a multistep biochemical process, involving a great number of different intermediates and bacterial groups, it is difficult to know the exact kinetics of each intermediate step. Therefore, a simplified procedure, assuming that only one reaction is rate limiting, is often applied to describe the kinetics of the whole process. For more complex substrates, this step is often the hydrolysis, for which a first order equation (Eq. (2)) is commonly used (Shahriari et al., 2012).

$$\mathrm{BMP}(t) = \mathrm{BMP}_\infty(1 - \exp(-k \times t)) \quad (2)$$

For some substrates, a second order equation (Monod-type alternative) is more suitable to describe the degradations kinetics as this allows for a prolonged slower degradation phase at the end of the process (Koch and Drewes, 2014; Eq. (3)).

$$\mathrm{BMP}(t) = \mathrm{BMP}_\infty\left(\frac{k' \times t}{1 + k' \times t}\right) \quad (3)$$

For substrates where two separate degradation profiles are apparent, often due to one more and one less readily degradable part, a combination of two first order equations (Eq. (4)) can be applied (Rincón et al., 2010).

$$\mathrm{BMP}(t) = \mathrm{BMP}_\infty(1 - X \times \exp(-k_1 \times t) - (1 - X) \times \exp(-k_2 \times t)) \quad (4)$$

In Eqs. (2)–(4), BMP_∞ is the ultimate BMP, BMP(t) is the BMP value at time t and k and k' are the rate constants for the first order and second order equations, respectively. In addition, in Eq. (4), X is the fraction of the more readily degradable part of the investigated substrate, k_1 is the rate constant of the same part whereas k_2 represents the rate constant of the less readily part.

2.2. Factors influencing volumetric gas measurements in BMP tests

In this section, some common factors that impact the accuracy of the gas quantification are presented. The factors are discussed with regards to their effect on measurements based on water displacement, also considering that the carbon dioxide is removed prior to the measurement.

2.2.1. Temperature and pressure

Since biogas is a compressible medium, the volume of the measured gas is highly dependent on the temperature and pressure. Normally, the volume is corrected to standard temperature and pressure using the ideal gas law. However, a number of different standard conditions are commonly accepted, which can lead to differences of up to 10% in the reported volumes (Walker et al., 2009). Thus, it is very important to clearly state which standard temperature and pressure was used when reporting the results. Furthermore, it is not just important that the pressure of the biogas is measured intermittently; for accurate and exact gas flow measurements, allowing the derivation of an exact dynamic profile of the gas flow, it should be monitored continuously. In fact, the ambient pressure can vary from day to day and without continuous monitoring of the gas pressure, valuable information of both the gas production dynamics and the accumulated volume could be lost. Eq. (5) shows how to adjust a gas volume to standard temperature and pressure based on the ideal gas law.

$$V_{STP} = \frac{p_{gas}}{p_{STP}} \times \frac{T_{STP}}{T_{gas}} V_{gas} \quad (5)$$

In Eq. (5), V_{STP} is the volume adjusted to standard temperature and pressure, p_{STP} is the standard pressure, p_{gas} is pressure of the measured gas, T_{gas} is the temperature of the measured gas in Kelvin (K), T_{STP} is the standard temperature in K and V_{gas} is the measured gas volume.

2.2.2. Water vapour

Biogas produced by anaerobic digestion is assumed to be saturated with water vapour and, in order to give accurate and precise quantitative gas measurements, the effect of the water should be minimised (Walker et al., 2009 and VDI 4630). At the ranges where anaerobic digestion tests normally are performed, (i.e. 0.6–1.1 bar and 10–40 °C) the vapour pressure of water can be satisfactorily approximated using the Antoine equation (Eq. (6)). It should be emphasised that, since the gas is measured at ambient temperature and pressure, it is these values that should be used and not the ones inside the reactor. As seen in the left graph of Fig. 1, the water content generates over-estimations of 2–8% in the gas volume at the normal ambient temperature range.

$$p_{vap} = 10^{8.1962 - \frac{1730.63}{T_{gas} - 39.724}} \quad (6)$$

In Eq. (6), p_{vap} is the water vapour pressure (mbar) and T_{gas} is the temperature (K) of the gas.

Another equation that has been used to describe the water vapour pressure in biogas is the Goff and Gratch equation (Walker et al., 2009). However, as seen in Fig. 1 (left graph), the Antoine equation provides an almost identical approximation (<0.2 mbar difference) as the Goff and Gratch equation of the vapour pressure at the temperature interval of interest and is thus more preferable to use due to its more simple nature.

2.2.3. Reactor headspace volume and composition

For cases when only the volume of methane is measured, using volumetric measuring principles, over-estimation of the gas production can occur when a reactor is flushed (de-aerated) with an inert gas (nitrogen gas is commonly used). As the biogas is produced it will mix with the flush gas and push away a mixture of the two gases. When the gas mixture reaches the carbon dioxide fixing unit, only the fraction of carbon dioxide will be absorbed, whereas the inert part of the flush gas will continue to the measurement unit and be registered. Thus, if less carbon dioxide is present in the flush gas compared to the produced biogas, this may lead to an over-estimation. To avoid this problem, a more expensive flush gas with the expected ratio of carbon dioxide and inert component could be used. However, if such a gas is utilised, it is important that no active components, affecting the process in anyway, are present in the gas. As an example, many standard gas compositions include relatively high concentrations of hydrogen, which could influence the degradation pathways and thus cause deviations in the gas production (Luo et al., 2012). Nevertheless, a simpler and cheaper method is to introduce a correction factor for the effect of the flush gas. Below follows an

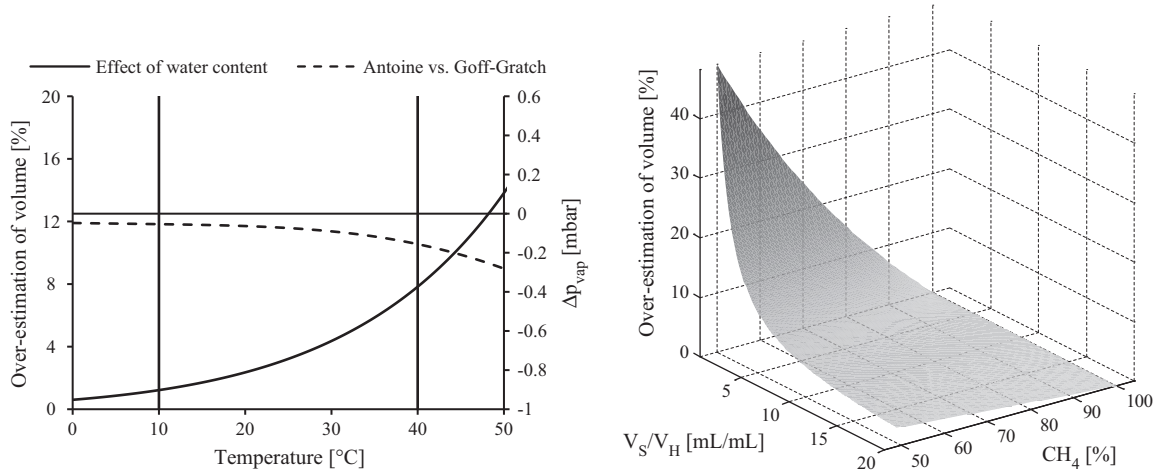

Fig. 1. Over-estimation of methane volume from the water vapour content in the biogas and the difference in predicted vapour pressure between Antoine and Goff–Gratch equation as a function of temperature (*left graph*). Over-estimation of methane volume as a function of the gas production to headspace ratio and methane content (*right graph*). Symbols: V_S – produced volume of methane V_H – headspace volume of bottle.

example of how such a factor can look like. It assumes that the over- or under-estimated volume at each measurement point is the amount of inert flush gas that is replaced by carbon dioxide in the headspace, and this can be expressed by Eq. (7):

$$V_{OE,i} = (x_{FG,i-1} - x_{FG,i}) \times V_H \times (X_{B,CO_2} - X_{FG,CO_2}) \quad (7)$$

In Eq. (7), $V_{OE,i}$ is the over-estimated volume at the specific measurement point i ($i \geqslant 1$), $x_{FG,_i}$ is the flush gas fraction in the headspace at the specific measurement point, V_H is the headspace volume, whereas X_{B,CO_2} and X_{FG,CO_2} are the carbon dioxide fractions in the produced biogas and flush gas respectively. If the gas mixture inside the reactor is assumed to be homogenously mixed then $x_{FG,i}$ can be calculated according to Eq. (8):

$$x_{FG,i} = x_{FG,i-1} - \frac{V_M \times x_{FG,i-1}}{V_H} = x_{FG,i-1} \times \left(1 - \frac{V_M}{V_H}\right) = \left(1 - \frac{V_M}{V_H}\right)^i \quad (8)$$

In Eq. (8), V_M is the volume for each measurement point. By combining Eqs. (7) and (8), the over-estimated volume can be expressed by Eq. (9):

$$V_{OE,i} = V_H \times (X_{B,CO_2} - X_{FG,CO_2}) \times \left(\left(1 - \frac{V_M}{V_H}\right)^{i-1} - \left(1 - \frac{V_M}{V_H}\right)^i\right) \quad (9)$$

Fig. 1 (right graph) presents a graph showing the response surface of the over-estimation in registered volume of gas as a function of the ratio between total gas volume and the headspace volume as well as the methane content of the biogas. As seen in the figure, the over-estimation increases greatly at low methane contents and when the totally produced gas volume is small in relation to the reactor's headspace volume. Up to 50% error, which might be even higher at lower methane contents, could be introduced by this factor.

2.2.4. Gas solubility

The solubility of gas components in the barrier solution of a measurement device following the liquid displacement procedure is another factor that could influence the results. Especially carbon dioxide is sensitive to this phenomena as its solubility in water at 25 °C is approximately 25 times higher compared to methane (Aylward and Findlay, 2002). The solubility can be decreased by increasing the salinity or decreasing the pH of the barrier solution (Müller et al., 2004; Walker et al., 2009). However, these procedures do not solve the problem entirely and create a rather unfriendly environment in the measurement unit. As this study only focuses on the measurement of methane gas, where the carbon dioxide has been removed beforehand, the problem of gas solubility is regarded as a smaller problem at the investigated flow rates. This factor was therefore not included in this study.

3. Experimental section

3.1. Equipment

The Automatic Methane Potential Test System II (AMPTS II, Bioprocess Control Sweden AB) was used for the BMP analysis. The AMPTS II is a standardised laboratory set-up specially designed for automatic BMP determination of any biodegradable material. The gas is measured through water displacement using flow cells that give a signal for approximately every 10 mL of produced gas. Temperature and pressure sensors are used to normalise the gas volume to 0 °C, 1 atm and dry gas conditions at each measurement point.

For the analysis of biogas composition, gas samples were collected at the end of the fermentation process with a gas-tight syringe and analysed using a gas chromatograph (GC) equipped with a thermal conductivity detector (TCD) and a metal column packed with a molecular sieve 5A 80/100 Mesh (Clarus 400, Perkin Elmer, USA).

3.2. Materials

The investigated samples in this study were one positive control substrate, i.e. microcrystalline cellulose (Cat. No. 166-142042, Fischer Scientific) and two, more traditional, biomass samples, i.e. garden waste from topped branches of *Clematis* and a lipid rich waste (i.e. old whip cream). Additionally, an anaerobic sludge (*i.e.* content from an anaerobic digester) collected from a sewage treatment plant in Sweden (Ellinge sewage plant, Sweden), which receives municipal wastewater and vegetable residues from the food industry, was investigated. This sludge was also used as inoculum and stored at room temperature for five days to reduce its organic content before using it in the experiment. No additional external nutrients or trace elements were added to the reactors before starting the BMP tests.

Sodium hydroxide (reagent grade 97%, pellets, Cat. No. 221465, Sigma–Aldrich) was used for the preparation of 3 M alkaline

solution for CO_2 fixation. A 0.4% Thymolphthalein pH indicator solution was prepared by dissolving the dye powder (2′,2″-Dimethyl-5,5″-di-iso-propylphenolphthalein, dye content 95%, Cat. No. 114553, Sigma–Aldrich) in a mixture containing 10% water and 90% ethanol (ACS reagent 99.5%, Cat. No. 459844, Sigma–Aldrich). N_2 gas (Air Liquid Gas AB, Kungsängen, Sweden) was used to obtain anaerobic conditions during the sample preparation phase.

3.3. Method description

3.3.1. Literature study

A short literature study was performed to observe how the gas normalisation in BMP tests has been reported in the literature. To limit the scope of the analysed papers, the study was exclusively focusing on digestions of cattle manure. The reported BMP values were ordered in groups based on the detail levels of the normalisation procedure presented in the paper. These values were then corrected (0 °C, 1 atm and dry conditions) to compensate for the lacking information, using an assumed temperature and pressure of 22 °C and 1 atm, if not otherwise stated, as the ambient experimental conditions.

3.3.2. Experimental design

In order to study the individual and combined effects of disregarding the four environmental factors on some key parameters in BMP tests (i.e. accumulated volume, BMP value and kinetic constants for a first and a second order equation as well as a model based on two combined first order equations), an experimental plan based on a 2^4 full factorial design, with 16 possible combinations, was implemented (Table 1). The proposed factorial design was used to evaluate the effect of the environmental factors in two ways: (*i*) by considering the degradation of different samples at one location and (*ii*) degradation of one sample at different geographic locations with varying ambient temperature and pressure. In order to evaluate the significance of the investigated factors, the relative changes introduced by neglecting one to three factors at the time were compared with the relative standard deviation of the measurements by applying different types of significance tests ($p > 0.05$) (for more information see Section 3.4 Numerical calculations). The kinetic constants were calculated with data sampled every quarter of an hour and the standard conditions used for the normalisation calculations were 0 °C, 1 atm, dry gas and the same initial headspace gas composition as the produced biogas.

Sample type: Four different sample types (i.e. anaerobic sludge, garden waste, microcrystalline cellulose and a lipid rich waste) were studied. These were chosen based on their different gas producing potentials and kinetic degradation profiles, giving a wider spread of data.

Geographic location: The environmental factors' effect on the results obtained from a BMP test of microcrystalline cellulose, performed at geographical locations with different ambient temperature and pressure conditions, was investigated. For this purpose, four model cities were chosen based on different altitudes (sea level or high altitude) and temperatures (warm or cold climate). The environmental conditions for the investigated locations are presented in Table 2. In order to simulate the effect of performing the tests at the investigated sites, the volumetric data from the BMP test performed in Lund, was adjusted for the temperature and pressure (adjusted to ambient pressure using the barometric formula) at each specific location according to the ideal gas law.

3.3.3. BMP test

The BMP test was performed with AMPTS II according to the description given in Section 2.1. Triplicates of each sample were used and the biological process was performed in standard 500 mL glass flasks, having a liquid volume of 400 mL, at 37 °C with continuously mixing of approximately 80–100 rotations per minute. The carbon dioxide was removed using 80 mL of 3 M sodium hydroxide solution for each reactor. The test was performed at roughly 80 m above sea level in a temperature controlled laboratory in Lund, Sweden. Temperature and pressure data from the test is presented in Table 2 (Lund) and some sample specific characteristics and other experimental related conditions are given in Table 3.

3.4. Numerical calculations

The accumulated volumes, reported in this study, were calculated by a cumulative summation of the adjusted measurement volumes for each cell opening registered by the measurement device. Each added volume was compensated for pressure, temperature, water vapour and headspace gas composition according to Eq. (10). The same equation was used in the calculations when neglecting one or more of these factors by setting the temperature and/or pressure ratio to 1 when neglecting the corresponding parameter, setting $p_{vap,i}$ to 0 when neglecting water vapour and, finally, setting $V_{OE,i}$ to 0 when neglecting composition of the headspace gas.

$$V_{acc,i} = V_{acc,i-1} + (V_M - V_{OE,i}) \times \left(1 - \frac{p_{vap,i}}{p_{gas,i}}\right) \times \frac{p_{gas,i}}{p_{STP}} \times \frac{T_{STP}}{T_{gas,i}} \qquad (10)$$

All calculations were performed with MATLAB® (MathWorks) and the rate constants were calculated using non-linear optimisation (*fmincon*) with a least square cost function. In order to reduce the risk of finding local minima, a multistart application, with 200 different initial parameter value sets, was applied. Furthermore, to avoid unrealistically low BMP values, the lower boundary of BMP_∞ was always set as the maximal BMP value of the raw data.

The effect of each factor included in the factorial design was calculated as the average of the difference between data points with and without considering the specific factor (Miller and Miller, 2010). In order to have comparable results, these are presented as the relative difference compared to the reference case (all factors considered). No interaction effects are presented as these were found to be close to zero for all cases. The significance level

Table 1
Factorial design of the experiment involving temperature (*T*), pressure (*p*), water vapour (*W*) and headspace gas composition (*H*) as factors. The two levels are designated by (–) when the factor is considered and a letter when the factor is disregarded in the normalisation.

Factor	Symbol	Factorial design															
		1[a]	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
Temperature	T	–	T	–	–	–	T	T	T	–	–	–	T	T	T	–	T
Pressure	p	–	–	p	–	–	p	–	–	p	p	–	p	p	–	p	p
Water vapour	W	–	–	–	W	–	–	W	–	W	–	W	W	–	W	W	W
Headspace composition	H	–	–	–	–	H	–	–	H	–	H	H	–	H	H	H	H

[a] Reference (all factors considered).

Table 2
Temperature and pressure data and time period, collected from Weather underground (2013), of the four geographical locations.

Conditions	Location	Altitude (m)	Temperature (°C)			Pressure [mbar]			Time period
			Mean	Min	Max	Mean	Min	Max	
Sea level/Cold	Lund	76	23.2 ± 0.3[a]	22.3[a]	23.9[a]	1016 ± 5	996	1036	10 Sep – 15 Oct 2013
Sea level/Warm	Bangkok	2	31.9 ± 1.3	29	34	1007 ± 2	1004	1010	20 Mar – 24 Apr 2013
Very high altitude/Cold	La Paz	4058	23.2 ± 0.3[a]	22.3[a]	23.9[a]	634 ± 2	631	637	10 Sep – 15 Oct 2013
High altitude/Warm	Kabul	1791	26.1 ± 2.2	22	29	827 ± 1	824	830	21 Jul – 25 Aug 2013

[a] Values from temperature controlled room.

Table 3
Data related to the BMP tests of the studied samples. The mean values are given together with the standard deviation. Symbols: *VS* – volatile solids concentration, $m_{I,S}$ – VS amount of inoculum in sample bottle, $m_{S,S}$ – VS amount of substrate in the sample bottle, V_H – headspace volume, X_{B,CO_2} – fraction of carbon dioxide in the biogas, X_{FG,CO_2} – fraction of carbon dioxide in flush gas, and V_S – produced volume of methane.

Sample	VS (%)	$m_{I,S}$ (g_{VS})	$m_{S,S}$ (g_{VS})	V_H (mL)	X_{B,CO_2} (%)	X_{FG,CO_2} (%)	V_S (NmL)	BMP (NmL/g_{VS})
Anaerobic sludge	1.1 ± 0.0	0	4.55[a]	240	26	0	310 ± 24[a]	68 ± 5
Garden waste	38.5 ± 2.9	4.45	2.60	240	26	0	805 ± 38	194 ± 29
Cellulose	96.2 ± 0.2	4.50	2.26	240	28	0	1166 ± 36	380 ± 16
Lipid rich waste	54.6 ± 4.6	4.48	2.25	240	20	0	1981 ± 56	743 ± 5

[a] Used as blank ($m_{I,B}$, V_B) in the BMP calculations of the other three samples.

($p = 0.05$) of the effects were determined by comparing their mean square error with the residual square error of the data set (Miller and Miller, 2010). In contrast, the significance level ($p = 0.05$) of the scenario combining the effects from all factors was determined with a Student's *t*-test (vs. reference). The performance of the BMP models was evaluated based on the Root Mean Squared Error of (RMSE) and the coefficient of determination (R^2), both calculated according to standard definition.

4. Results and discussion

4.1. Gas normalisation in the literature

As seen in Table 4, only one of the 23 studied papers reported that the gas volume was corrected for temperature, pressure and water vapour. Eight reported a correction for temperature and pressure but not for water vapour, whereas seven were missing information regarding correction for any of them. Four of the papers lacked information about the gas normalisation and whether a blank control or pre-incubation of the inoculum was performed in order to reduce the influence of the inoculum's own gas production. After correcting for the missing information, the difference in average BMP was reduced from 192–223 NmL/g_{VS} to an interval of 186–201 NmL/g_{VS}. The papers missing information about compensation for inoculum gas production has a slightly higher average BMP (i.e. 201 NmL/g_{VS}), which may be explained by this omission.

It should also be mentioned that cattle manure is a substrate that can vary substantially in quality, illustrated by the large standard deviations seen in Table 4, and, given this variability, the results should be regarded with much caution. Many more data points are needed to be able to draw any definitive conclusions. In this context, the low average value (100 NmL/g_{VS}) for the only study correcting for all factors cannot be considered representative as more data is required for a reliable mean. Furthermore, the process of ordering the studied references was strictly based on what information that could be found in the papers. It is therefore not certain whether this was correctly done, as this information might have been omitted and, as a consequence, misinterpreted as non-normalised. Another potential source of error is the fact that many groups have used a liquid displacement based technique

Table 4
Results from literature search focused on BMP tests involving cattle manure as a substrate. The records are sorted according to the level of detail in the normalisation step (0 °C, 1 atm and dry gas) given in the reference. Symbols: *T* – ambient temperature, *p* – ambient pressure, *W* – water content in the gas, and *I* – consideration of gas production from inoculum.

	BMP raw (mL/g_{VS})	BMP adjusted (NmL/g_{VS})	Reference		BMP raw (mL/g_{VS})	BMP adjusted (NmL/g_{VS})	Reference
All factors considered				*No W T p*			
	100	100	Chen et al. (1988)		233	210	Lehtomäki et al. (2007)
Mean	100 ± 0	100 ± 0			218	197	Sutaryo et al. (2012)
No W					144	130	Li et al. (2009)
	212	207	Seppälä et al. (2013)		140	126	Qiao et al. (2011)
	238	232	Luna-delRisco et al. (2011)		240	216	Krishania et al. (2013)
	144	140	Amon et al. (2007)		238	215	Umetsu et al. (2006)
	241	235	El-Mashad and Zhang (2010)		230	207	Otero et al. (2011)
	111	108	Chen et al. (2010)	Mean	206 ± 44	186 ± 40	
	206	201	Demirbas (2006)	*No W T p I*			
	51	50	Li et al. (2013)		235	212	Crolla et al. (2011)
	330	321	Risberg et al. (2013)		120	108	Guliano et al. (2013)
Mean	192 ± 87	187 ± 85			307	277	Rico et al. (2007)
Uncertain					230	207	Luste et al. (2012)
	190	190	Møller et al. (2004)	Mean	223 ± 77	201 ± 70	
	177	177	Wang et al. (2012)				
	90	90	Lisboa and Lansing (2013)	*Overall*			
Mean	152 ± 54	152 ± 54		Mean	192 ± 70	181 ± 64	

for quantifying the raw biogas. As the carbon dioxide was not removed, there is a high possibility that the gas production was under-estimated, due to solubilisation of this gas in the barrier solution, in these cases (Walker et al., 2009). However, despite all uncertainties, the obtained results still serves a purpose to illustrate the problem in data evaluation that arises when this kind of information is not properly given.

4.2. Different samples analysed at one location

The normalised BMP curves, calculated using Eqs. (1) and (10), for anaerobic sludge, garden waste, cellulose and the lipid rich waste are presented in Fig. 2. The BMP values and first order rate constants from these curves were used as reference for evaluating the effects (relative difference vs. reference) coming from neglecting one or more of the gas normalisation factors. The final BMP values together with all the experimental conditions used in the calculations are presented in Table 3. As seen, the four samples generated different BMP values, providing a widespread foundation for the evaluation.

When factors like temperature, pressure, water vapour and/or headspace gas composition are neglected, their effects on the calculated accumulated methane volume, BMP values and rate constants are evident, and can be expressed as the relative difference compared to a correctly normalised case as presented in Fig. 3. Presented here are also the relative standard deviations (RSD, $n = 3$) from the BMP test of each substrate. As seen, the variations in accumulated volume and BMP from the experimental measurements are decreasing with high gas potential and homogenous characteristics of the substrates. The largest effect of a singular factor is achieved for anaerobic sludge when the headspace gas composition is neglected. This can be explained by the low gas production during the degradation of this sample. A comparison of the effects on the calculated BMP and accumulated volume of the other three samples shows that the impact from neglecting the headspace gas composition is smaller with regard to the BMP values. The reason for this is that a part of the over-estimation, coming from the difference in headspace gas, is removed when the BMP value is adjusted for the gas production of the inoculum. In fact, for the lipid rich waste, neglecting this factor introduced a small under-estimation due to the higher methane content in the gas. With regard to all samples, temperature is the most substantial contributor with relative effects slightly below 10% in both accumulated volume and BMP value. The lone factor introducing a reduction, compared to the reference value, is the pressure, which can be explained by the slightly higher average pressure vs. the atmospheric one (Table 2). Combining all factors leads to significant effects for all sample types with a maximal effect close to 30% for anaerobic sludge sample. A statistical analysis shows that the effect from temperature and all factors combined on the BMP value is significantly higher ($p > 0.05$) compared to the experimental results obtained for all substrates except garden waste, which is characterised by high heterogeneous properties. With regard to the lipid rich waste even the effects from neglecting the water vapour content and ambient pressure are significant. The fact that the errors coming from neglecting the environmental factors many times are larger compared to the experimental variations demonstrates the importance of considering these parameters for reliable and comparable results.

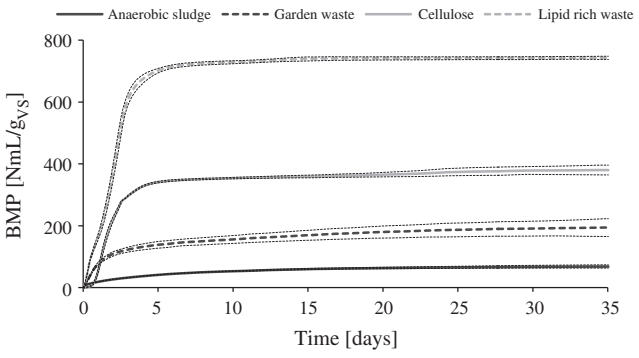

Fig. 2. BMP curves for the four investigated sample types, i.e. anaerobic sludge, garden waste, cellulose and lipid rich waste. The dotted lines around the degradation curves represent the standard deviation.

As seen in Table 5, which presents the calculated first order rate constants for the four sample types together with the RMSE and R^2 of the evaluated equations, the standard deviations from the rate constants are rather large. This is a clear indication that the kinetics of the process is highly sensitive to other factors, unrelated to the ones investigated in this study. Furthermore, the large RMSE and small R^2 values for the first order equation demonstrate a rather poor fit for this model. With regard to anaerobic sludge and garden waste, a slightly better fit is observed for the second order vs. the first order equation. However, the two combined first order equations was the best fitting model. None of the models provides satisfactory predictions for cellulose or the lipid rich waste. This illustrates the diversity of the kinetic profiles from anaerobic degradation of different sample types. Therefore several model types should be tested and evaluated for each substrate in order to find the most suitable one.

The relative effects on the calculated rate constants, presented in Fig. 3, reveal that neither temperature, pressure nor water vapour have a noteworthy impact on the kinetic calculations. This shows that, not only the average temperature and pressure values, but also the variations in these parameters, have a small impact on the kinetic calculations. Neglecting the initial headspace gas composition, on the other hand, leads to noticeable over-estimations with up to 10% deviations in the calculated k, k' and k_1. Similarly to the BMP value, the effects from neglecting the headspace gas composition seem to be less pronounced when the total gas production increases. More varying effects are seen on k_2 for the combined first order equation. However, only on two occasions are the effects on the calculated rate constants significant. Thus, even under close to identical experimental conditions, the biological factors have a larger impact than the studied gas normalisation factors. However, in contrast to the random error coming from the biological variability, neglecting the headspace gas composition will introduce a systematic error, which always should be avoided.

4.3. One sample analysed at different geographical locations

The effect of performing BMP tests under extreme environmental conditions prevailing in different geographical locations, was investigated by simulations of earlier obtained data with the investigated site's temperature and pressure profiles. As seen in Fig. 4, showing the factorial design effects on accumulated methane volume and BMP value, the effects from not considering the investigated factors are in general larger as compared to the experimental variation regardless of location. The largest error would be recorded in La Paz followed by Kabul, both located at higher altitudes showing that the lower atmospheric pressure at these locations clearly has a profound effect on the volumetric measurements. It should also be noted that, in addition to the gas normalisation aspects, a lower atmospheric pressure might also affect the biology of the process as it changes the solubility of gases in the liquid. Previous studies have come to different conclusions regarding this aspect. Alvarez et al. (2006) found no significant effect between running a continuous reactor at 495 or 760 mmHg. Jiang et al. (2010), on the other hand, observed a negative effect

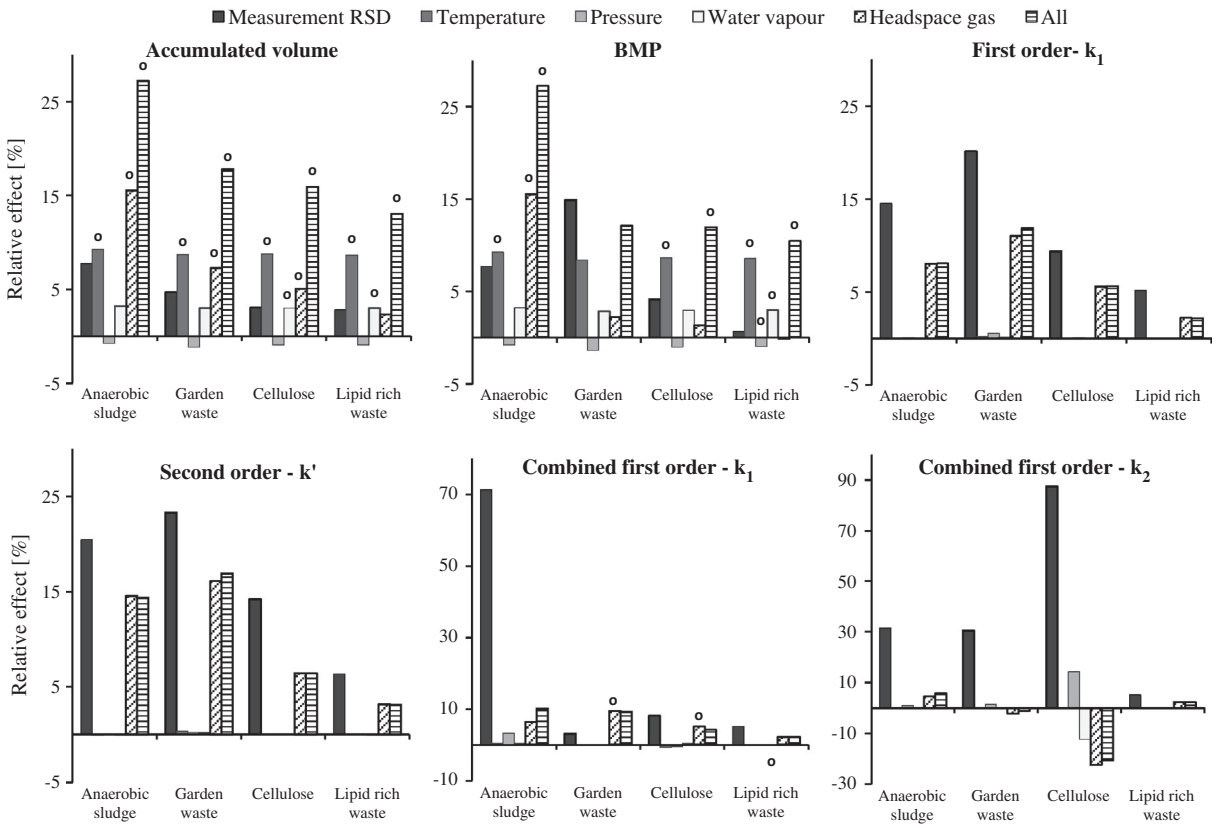

Fig. 3. Relative effects (vs. reference) from the evaluated factors on accumulated volume, BMP value and calculated rate constants for different samples. The first column of each sample presents the experimental relative standard deviation (RSD) between replicates ($n = 3$). A circle (o) above the column represents a significant effect.

Table 5
Calculated rate constants and fitting parameters for the studied model types. The mean values are given together with the standard deviation. Symbols: k, k', k_1 and k_2 – rate constants of respective model, *RMSE* – root mean squared error, and R^2 – coefficient of determination.

Sample	First order kinetics			Second order kinetics			Two combined first order kinetics			
	k (1/day)	RMSE (NmL/g_{VS})	R^2 (–)	k' (1/day)	RMSE (NmL/g_{VS})	R^2 (–)	k_1 (1/day)	k_2 (1/day)	RMSE (NmL/g_{VS})	R^2 (–)
Anaerobic sludge	0.17 ± 0.03	2.9 ± 0.7	0.99	0.25 ± 0.05	1.1 ± 0.1	0.99	1.58 ± 1.13	0.12 ± 0.04	0.6 ± 0.2	1.00
Garden waste	0.23 ± 0.07	14.9 ± 4.4	0.92	0.48 ± 0.11	5.7 ± 2.4	0.97	0.78 ± 0.30	0.19 ± 0.21	1.1 ± 0.1	1.00
Cellulose	0.40 ± 0.06	18.8 ± 3.9	0.95	0.66 ± 0.09	21.6 ± 0.8	0.92	0.44 ± 0.03	0.27 ± 0.24	16.1 ± 1.9	0.96
Lipid rich waste	0.45 ± 0.02	25.9 ± 2.1	0.97	0.71 ± 0.05	45.6 ± 2.3	0.90	8.52 ± 14.0	0.45 ± 0.02	25.9 ± 2.1	0.97

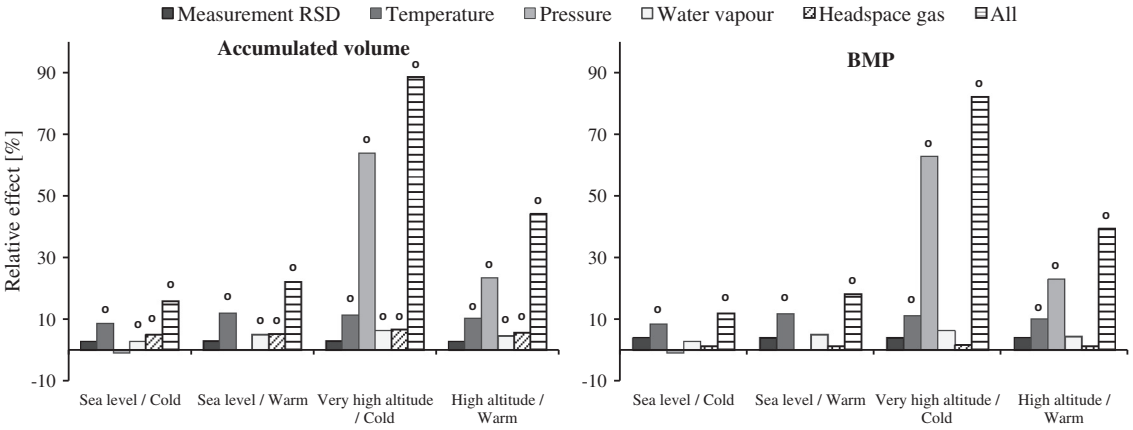

Fig. 4. Relative effects (vs. reference) from the evaluated factors on accumulated volume and BMP value of cellulose when the analysis takes place at different geographical locations. The first column of each sample presents the experimental relative standard deviation (RSD) between replicates ($n = 3$). A circle (o) above the column represents a significant effect.

at medium organic loading rates, positive effects at higher organic loading rates and no effect on lower organic loading rates when comparing operation at 658 vs. 1010 mbar. In comparison to the pressure, the temperature has a smaller, but still significant effect. This is most profoundly shown by the 12% effect registered for Bangkok, having an average temperature above 30 °C. The effects from water vapour and headspace gas composition are smaller compared to temperature and pressure but still have a substantial contribution as they have an additive effect. These results clearly demonstrate the importance of considering the temperature and pressure when performing BMP tests at locations with extreme environmental conditions.

5. Conclusions

Based on the results of this study it can be concluded that, compensating for temperature, pressure, water vapour and headspace gas composition is important for producing comparable standardised results from BMP tests. Adjusting for temperature and pressure is particularly important when tests are performed at locations with more extreme environmental conditions, especially for laboratories at high altitudes. It can also be concluded that neglecting these factors has a small impact on the kinetic calculations.

It should be stated that the effects coming from the factors studied in this work are only a few of the aspects that may influence the results. In fact, other parameters such as particle size, origin of inoculum, inoculum-to-substrate ratio, mixing rate, and process temperature, much likely have a considerable impact and are discussed in detail in other publications (Angelidaki et al., 2009; Raposo et al., 2011b).

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