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Addressing case specific biogas plant tasks: Industry oriented methane yields derived from 5 L Automatic Methane Potential Test Systems in batch or semi-continuous tests using realistic inocula, substrate particle sizes and organic loading

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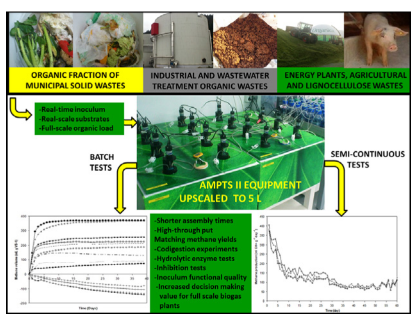
HIGHLIGHTS

- In-house upscale of Automatic Methane Potential Test System to 5 L was developed.
- Full-scale inocula, substrates and organic loading were used in tests.
- Tests were characterized by short preparation times, accuracy and high-through put.
- Same scales, equipment and methodologies were used in batch and continuous tests.
- Increased full-scale decision making value of the developed approach was shown.

GRAPHICAL ABSTRACT



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ABSTRACT

The primary aim of the study was to develop and validate an in-house upscale of Automatic Methane Potential Test System II for studying real-time inocula and real-scale substrates in batch, codigestion and enzyme enhanced hydrolysis experiments, in addition to semi-continuous operation of the developed equipment and experiments testing inoculum functional quality. The successful upscale to 5 L enabled comparison of different process configurations in shorter preparation times with acceptable accuracy and high-through put intended for industrial decision making. The adoption of the same scales, equipment and methodologies in batch and semi-continuous tests mirroring those at full scale biogas plants resulted in matching methane yields between the two laboratory tests and full-scale, confirming thus the increased decision making value of the approach for industrial operations.

1. Introduction

The biological methane potential (BMP) test is routinely used to experimentally determine quantities of methane produced per gram of volatile solids (ml CH_4 g^{-1} VS^{-1}) or per gram chemical oxygen demand (ml CH_4 g^{-1} COD^{-1}) (Angelidaki and Sanders, 2004; Lesteur et al., 2010) in a simple batch system under anaerobic con-

Abbreviations: AMPTS, automatic methane potential test system; COD, chemical oxygen demand; FM, fresh matter; HRT, hydraulic retention time; IWWS, industrial wastewater sludge; MWWTP, municipal waste water treatment plant; OLR, organic loading rate; OFMSW, organic fraction of municipal solid wastes; SPMS, secondary paper mill sludge; TIC, total inorganic carbon; TKN, total Kjeldahl nitrogen; TS, total solids; VOA, volatile organic acids; VS, volatile solids.

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ditions (Batstone and Jensen, 2011; Chynoweth et al., 1993; Owen et al., 1979). It has been used for designing, monitoring and operating full-scale anaerobic digestion and also for determining loading rates of less well characterized substrates and respective hydraulic retention times (Lesteur et al., 2010). However, in most BMP assays the tests are conducted on the scale of 0.5 L volumes or even smaller (Angelidaki et al., 2009; Batstone and Jensen, 2011; Raposo et al., 2011; VDI 4630, 2006). To achieve sufficient reproducibility and standardization the biomass and solid substrate particles used in batch tests are generally homogenized by filtration and/or mechanical disintegradtion, respectively, effectively shifting particle size distribution towards particles smaller than 5 mm (Carrere et al., 2010; Frigon et al., 2012; Gomez-Tovar et al., 2012; Herrmann et al., 2012b; Raposo et al., 2011).

Methane production from substrates such as food waste, sludge from wastewater treatment plants, paper mills, wheat or barley straw and other have been shown to increase significantly after the reduction of particle size by mechanical, chemical, combinatorial or enzymatic pretreatments (Lindmark et al., 2012; Menardo et al., 2012; Zhang and Banks, 2013). Although the positive effects on biogas production of such pretreatments are often obvious for some substrates, the net balance between the increase in methane yield relative to energy consumed for pretreatment is not, and depends on a number of industrial decision making and real-life parameters: pretreatment side effects, substrate identity, year to year variation in substrate chemical characteristics, particle size distribution, loading and mixing rates, inoculum characteristics, mechanical pretreatment machinery installation costs, operation and also wear and tear costs and biogas plant revenues (Herrmann et al., 2012a,b; Lindmark et al., 2012; Menardo et al., 2012; Zhang and Banks, 2013).

Consequently, the specific industrial viability of operations in particular biogas plant is in fact highly case specific and difficult to generalize from published data. In order to attain profitability through the compromise between energy/effort input in substrate modifications (e.g. particle size reduction or chemical pretreatment) and subsequent energy yields (Lauwers et al., 2013; Palmowski and Muller, 2003; Raposo et al., 2011) the biogas plant decision makers require data derived from site specific inoculum and substrates under conditions mirroring those in full scale reactors (Angelidaki et al., 2009). However, the systematic errors of methane potential test were recently reviewed and the lack of uniformity and multifactorial dependence limiting their direct industrial applicability illustrated (Angelidaki et al., 2009; Izumi et al., 2010; Palmowski and Muller, 2000; Walker et al., 2009; Raposo et al., 2011). In addition, generally lower methane yields were reported in continuous digestion than in batch tests (Mudhoo, 2012) due to the lack of uniformity in the experimental setup conditions and also differences in residence times.

The primary aim of the study was to develop and validate an in-house upscale of the commercially available Automatic Methane Potential Test System II (AMPTS II; Bioprocess Control, Sweden) (Badshah et al., 2012) from 0.5 to 5 L (optionally 10 L) for studying the use of real-time inocula from full scale digesters and real-scale substrates available on-site. Second, a number of locally available resources ($n = 20$; ($n_{solid} = 17$; $n_{liquid} = 3$)) of industrial relevance for biogas production were analyzed and their methane yields related to those found in published literature. Third, the extent of methane yield increase was tested (i) using two distinct industrial inocula receiving two different substrates and two commercially available enzyme additives; and (ii) using codigestion optimization of four substrates (secondary paper mill sludge (SPMS), industrial waste water sludge (IWWS), maize silage and organic fraction of municipal solid waste (OFMSW). Fourth, to fill the gap between batch and semi-continuous tests the developed equipment was also tested as semi-continuous 5 L pilot system in anaerobic degradation of SPMS. Last, functional quality of inocula derived from Sijanec, Vucja vas and Logarovci biogas plants was assessed by analyzing daily methane production rates from three real-scale lignocellulose substrates (maize silage, wheat bran, corn meal) in attempt to generalize the obtained results (Fig. S1).

2. Methods

2.1. The upgrade of Automatic Methane Potential Test System II (AMPTS II) to 5 l scale

Three sets of AMPTS equipment (Bioprocess Control, Sweden) (Badshah et al., 2012) for fast and accurate on-line measurements of ultra-low biogas and biomethane flows were used for determination of biogas potential of various substrates on laboratory scale due to its accuracy and high throughput. In this study, the 0.5 L AMPTS II glass reactors were replaced with 5 L reactors (Schott, Germany). Due to inability of AMPTS II temperature incubation system to host 5 L reactor vessels waterproof Styrofoam (Synthos XPS, Synthos Group) water baths with lids were manufactured in house instead (Fig. S2A). The water bath heating and mixing were achieved through professional heaters (3619 Aquarium Heater 300 W, Eheim Jäger, Germany) and submersible agitators (NWA 1.6 adj, Newave, China) to ensure uniform distribution of heat across the entire water bath that was independently verified using installed thermometers (38–55 °C). For convenience and handling efficiency two water baths with capacity of housing up to ten 5 L (or eight 10 L) reactors each were constructed for each AMPTS II system. Rotating shaft of computer controlled mechanical agitator was modified to fit larger bottles and provided comparable mixing of reactor content as originally designed (Fig. S2B).

The standard approaches (e.g. Angelidaki et al., 2009; VDI 4630, 2006) require biomass and solid substrate pretreatment, either as filtration and grinding to reduce substrate and biomass particle sizes significantly in addition to buffer amendments and dilutions of biomass and substrates. In order to identify and map the extent of methane yield mismatch in using 0.5 and 5 L batch scales, Vucja vas agricultural biogas plant reactor 1 (Table 1) was sampled immediately after its regular substrate amendment (10 g VS l^{-1}) and complete volume homogenization, representing the standard active biomass amended with regular substrate mix (Table 1). An aliquot of that homogenized sample was mechanically pretreated using knife mill (800 W Philips blender HR2096; 10.000 rpm, 5 min) to enable accurate particle size decrease. Seven replicates were used from the same inoculum batch to fill 0.5 and 5 L reactors with 0.4 and 4 L of mechanically pretreated and unmodified inoculum, respectively. A wet sieving analysis was carried out before and after mechanical treatment using 10, 3, 1 and 0.2 mm mesh sieves in six replicates.

To ensure gas tight conditions rubber stoppers were lubricated with silica gel before assembly and each reactor was flushed with N_2 for about 3 min before the device startup. The 15 min on–off mixing regime, organic loading (10 g VS l^{-1}) and mesophilic conditions (38 ± 2 °C) adopted from biogas plants were used (Table 1). The resulting methane yields were normalized to standard conditions as described before (Badshah et al., 2012).

All materials, equipment and procedures can be obtained from the authors upon request.

2.2. Biomass sampling

Three agricultural biogas plants Sijanec (Ormoz, Slovenia; 1 MW), Vucja vas (Bucecovci, Slovenia; 4 MW) and Logarovci (Logarovci, Sloveniaa; 1 MW) (http://keterorganica.com) and municipal waste water treatment plant (MWWTP) Velenje (150 kW; Velenje,