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Biswas, Rajib; Ahring, Birgitte Kiær; Uellendahl, Hinrich

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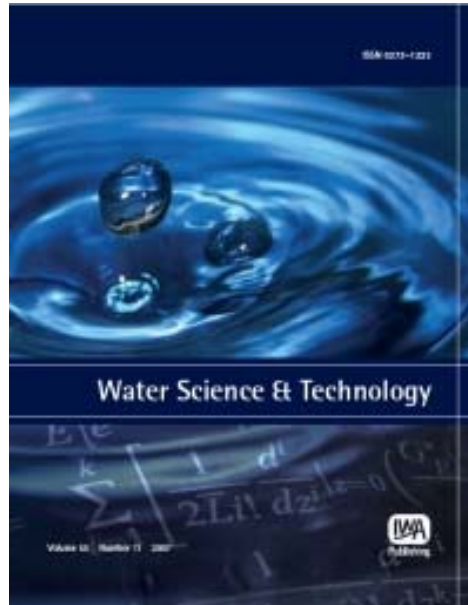
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Improving biogas yields using an innovative concept for conversion of the fiber fraction of manure

Rajib Biswas, B. K. Ahring and H. Uellendahl

ABSTRACT

The potential of a new concept to enable economically feasible operation of manure-based biogas plants was investigated at laboratory scale. Wet explosion (WEx) was applied to the residual manure fibers separated after the anaerobic digestion process for enhancing the biogas yield before reintroducing the fiber fraction into the biogas reactor. The increase in methane yield of the digested manure fibers was investigated by applying the WEx treatment under five different process conditions. The WEx treatment at 180 °C and a treatment time of 10 min without addition of oxygen was found to be optimal, resulting in 136% increase in methane yield compared with the untreated digested manure fibers in batch experiments. In a continuous mesophilic reactor process the addition of WEx-treated digested fibers in co-digestion with filtered manure did not show any signs of process inhibition, and the overall methane yield was on average 75% higher than in a control reactor with addition of non-treated digested fibers.

Key words | anaerobic degradation, digested manure fibers, methane yield, wet explosion treatment

Rajib Biswas
B. K. Ahring
H. Uellendahl (corresponding author)
Section for Sustainable Biotechnology,
Aalborg University Copenhagen,
Lautrupvang 15,
2750 Ballerup,
Denmark
E-mail: hu@bio.aau.dk

B. K. Ahring
Center for Bioproducts and Bioenergy,
Washington State University,
2710 Crimson Way,
Richland,
WA 99354,
USA

INTRODUCTION

Biogas produced from manure, organic waste and plant biomass is becoming increasingly attractive in terms of reducing greenhouse gas emission, nutrient recovery and well as renewable energy alternatives to fossil fuels. It was shown in a recent life cycle assessment that biogas production from manure has the highest reduction effect on greenhouse gas emissions compared with other biofuels production processes (Thyø & Wenzel 2007). Despite the environmental benefits, the economical operation of centralized biogas plants based on manure alone is difficult due to a low methane yield per volume unit of manure (Gerin *et al.* 2008). Thus manure-based biogas plants are currently depending on the co-digestion of industrial waste with a high methane yield, typically originating from the food industry. While for example 40 million tons of manure produced annually in Denmark represents a huge biogas potential, only 5% of this amount is currently treated in biogas plants, and the implementation of centralized biogas plants in Denmark has stagnated throughout the last 10 years due to the fact that the operation based on manure alone has not been viable and the availability of industrial organic waste is limited (Jensen *et al.* 2009). The Danish governmental program 'Green Growth' targets an

exploitation of up to 40% of manure in 2020 (Danish Government 2009), corresponding to an increase in biogas plant capacity by 8-fold, equivalent to more than 70 new centralized biogas plants of the largest scale. As manure will be the main substrate for these future biogas plants it is a prerequisite to achieve an economically feasible operation of manure-based biogas plants in order to see this program come into full implementation. For economic operation of a biogas plant, biogas yields of more than 30 m³ per m³ feed are needed to compensate for the transportation costs (Uellendahl *et al.* 2007). The biogas yield of manure in conventional biogas plants is often lower as the organic matter content in manure is typically less than 10%, of which 60–80% is fiber material, which leads to methane yields of only 30–50% of the methane potential (Hartmann *et al.* 2000; Christensen *et al.* 2007; Boe & Angelidaki 2009). Intensive research has been carried out for improving the biogas yield of manure and sludge by implementing a wide range of biological, chemical, mechanical, and thermal pretreatment methods (Angelidaki & Ahring 2000; Hartmann *et al.* 2000; Carrère *et al.* 2010), in co-digestion with organic waste (Angelidaki & Ellegaard 2003), in combination with solid–liquid separation of manure

(Mladenovska *et al.* 2006; Christensen *et al.* 2007; Møller *et al.* 2007; Kaparaju & Rintala 2008), and in different digester configurations (Boe & Angelidaki 2009; Kaparaju *et al.* 2009). Comparing the different treatment methods for increasing the biofuel yield from lignocellulosic biomass, it was found that thermo-chemical treatment was the most suitable for the treatment of lignocellulosic biomass for subsequent conversion into biogas (Angelidaki & Ahring 2000; Lissens *et al.* 2004; Uellendahl *et al.* 2007). Thermal hydrolysis has proven commercial viability for enhancing biogas production of sludge and household waste through the implementation of a number of large-scale plants worldwide by the company Cambi (Elliott & Mahmood 2007).

Wet explosion (WEx), a steam explosion process with or without addition of oxygen, has previously shown a high potential for the destruction of the lignocellulosic structure of biomass, in order to enable the hydrolysis for subsequent ethanol fermentation (Klinke *et al.* 2002; Lissens *et al.* 2004; Sørensen *et al.* 2008). Generally, WEx includes both physical disruption and a partly chemical degradation of the biomass (Sørensen *et al.* 2008). The WEx treatment equipment, patented by the Danish company Biogasol ApS, can handle material with up to 30% dry matter, and results in high sugar yields, which can subsequently be converted into ethanol or methane (Christensen *et al.* 2007; Ahring & Langvad 2008). Previous studies revealed that the effect of the WEx treatment is correlated to the content of lignin in lignocellulosic fiber material (Uellendahl *et al.* 2007). As a consequence, the combination of the WEx treatment

with biogas production from manure was evaluated to be most beneficial when applying the treatment to manure fibers separated from the effluent of a biogas reactor after digestion (Figure 1).

In the present research project called FiberMaxBiogas the potential of the new concept of combining the recirculation of the digested fiber fraction with the WEx treatment for increasing the biogas yield of manure is investigated. In the following we will present the first overall results from batch tests for screening of the increase of the biogas yield of the digested fiber fraction after WEx treatment and the long-term performance of adding the WEx-treated digested fibers in co-digestion with manure in laboratory-scale reactors.

MATERIAL AND METHODS

Digested fiber fraction

The effect of the WEx treatment was tested on digested fibers separated from the effluent of one of the biogas reactors of the Biokraft A/S centralized biogas plant on Bornholm, Denmark. For separation of the digested fiber fraction an industrial-scale decanter centrifuge was used. The Biokraft A/S biogas plant is operated under mesophilic conditions (38 °C) with a hydraulic retention time (HRT) of 20 days, treating manure (>90% vol.), co-digested with agricultural residues (<5% vol.) and industrial waste (<5% vol.) from food-processing industries.

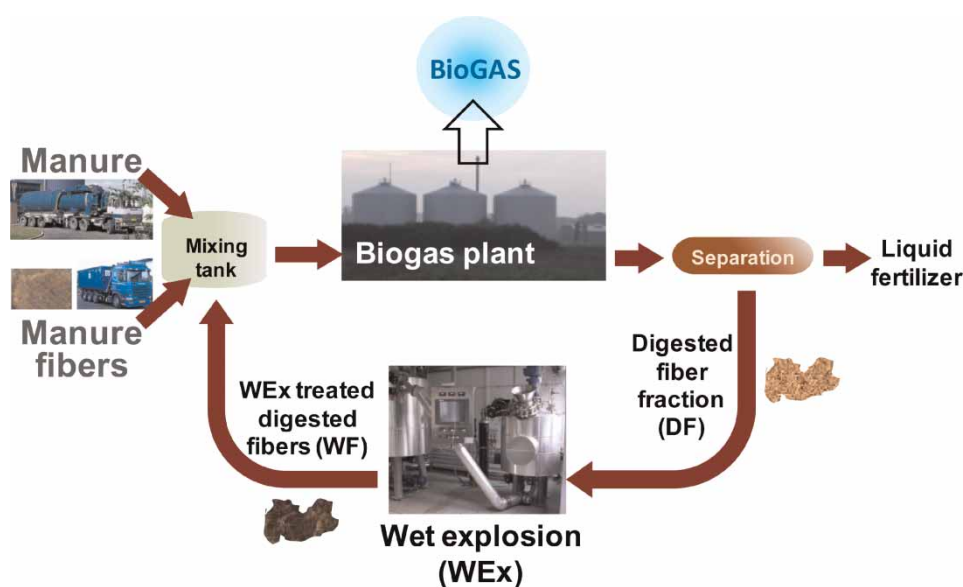


Figure 1 | Overview of the new manure-to-biogas concept with WEx treatment of the digested fiber fraction separated from the effluent.

Filtered manure

The digested fiber fraction was added to the laboratory-scale reactors in co-digestion with filtered cow manure (FCM). FCM was obtained from cow manure delivered to the biogas plant of Biokraft A/S filtered through 10 mm sieves in order to avoid clogging of the influent tube. Filtered mixed manure (FMM) was used for the co-digestion of the digested fiber fraction from day 138 to 180. FMM was obtained from a mixture of manure from cows, pigs and poultry in a ratio of 70:25:5 on a total solids (TS) basis.

Wet explosion treatment

Wet explosion treatment was performed in a laboratory-scale 3.5 L batch reactor with a maximum active volume of 2.0 L, provided by Biogasol A/S. The reactor is equipped with continuous stirring (990 rpm) a gas and liquid dosage system for supply of additives (H_2O_2 , H_2SO_4 , O_2 , Na_2CO_3 etc.), and a flush valve for sudden pressure release into a 25 L subsequent flash tank. The reactor is heated by an external oil heater. The denoted process temperature was the temperature measured at the reactor top.

Digested fibers were treated in 1 kg batches, adding 400 g of tap water to 600 g of fiber material for achieving a TS concentration of 12% inside the reactor. After the denoted treatment time in the reactor, the biomass was flushed into the flush tank. The different treatment conditions are displayed in Table 1. Heating times to reach the start temperature varied between 7 and 15 min due to the different final temperature.

Batch experiments

The methane yields of treated and untreated digested fibers were determined in laboratory-scale anaerobic batch tests using 117 mL vials under mesophilic condition ($38 \pm 0.5^\circ\text{C}$).

Inoculum for the batch experiments was supplied from one of Biokraft's biogas reactors and stored at 4°C . Before batch set-up, the inoculum was pre-incubated at 38°C for one week.

Two different inoculum-to-substrate ratios (ISRs) were tested, i.e., 1.0 g volatile solids (VS)/vial (ISR 1) and 0.5 g-VS/vial (ISR 2) of the substrate were added to vials with 25 mL inoculum. After filling with the respective biomass and inoculum, batch vials were flushed with N_2/CO_2 (80%/20%) prior to closing air tight with rubber stoppers and aluminum crimps. Experimental set-up was performed in triplicate and a triplicate of vials filled with 25 mL inoculum and water instead of substrate was used as control. The vials were incubated until no significant further biogas production was detected (48 days). The methane yield of the treated and untreated digested fibers was determined by measuring the methane concentration in the headspace using gas chromatography (GC) (SRI-GC-310) and calculated according to Equation (1). Overpressure in the vials was released whenever necessary and the methane concentration in the headspace was determined before and after the gas release for calculation of the cumulative methane yield. Methane production in the controls filled with inoculum only was subtracted to calculate the methane yield from the added substrate ($\text{mL/g-VS}_{\text{added}}$). A gas mixture of CH_4/N_2 (30%/70%) was used as standard gas mixture for gas GC.

$$\text{CH}_4\text{yield}_S = (\text{CH}_4\%_S \cdot V_{\text{headspace},S} - \text{CH}_4\%_C \cdot V_{\text{headspace},C}) / \text{g-VS}_{\text{added},S} \quad (1)$$

where index S: added substrate; index C: control vials (without substrate).

CSTR experiments

Two 5 L stainless steel continuous stirred tank reactors (CSTRs) with a working volume of 3 L were operated with a HRT of 20 days. The reactor temperature was maintained at $38 \pm 0.5^\circ\text{C}$ by circulating hot water in the heating jacket using a water bath. In order to evaluate the biogas process of WEx-treated digested fibers, CSTR experiments were performed by feeding WEx-treated digested fibers (WF) in a test reactor (R1) and non-treated digested fibers (DF) in a control reactor (R2). The fiber fraction was in both reactors co-digested together with FCM. The WF were pretreated at 180°C for 10 min. Both reactors were fed (150 mL) twice a day using peristaltic pumps (Watson-Marlow 610 series). The produced biogas was registered using volumetric gas

Table 1 | WEx conditions tested for the treatment of digested manure fibers (DF)

Batch	Treatment time (min)	Temperature ($^\circ\text{C}$)	Pressure (bar)	Addition of O_2 (bar)
145-10	10	145	2.2	–
165-10	10	165	3.3	–
165-20	20	165	7.4	–
165-10- O_2	10	165	12.4	6
180-10	10	180	9.9	–

meters, logging the gas production automatically in 10 mL intervals. Reactors were stirred for 5 min 10 times a day. The performance of the reactors was monitored on the basis of methane yield, volatile fatty acids (VFAs) concentration and pH.

During start-up, both reactors R1 and R2 were filled with 3 L of inoculum, originating from one of Biokraft's biogas reactors. The feeding was started with FCM alone (days 0–54) with an organic loading rate (OLR) of $2.5 \text{ g} \cdot \text{VS}/(\text{L} \cdot \text{d})$ and a HRT of 20 days. On day 55, co-digestion of the DF with FCM was initiated in R1 and R2 at a feed ratio of 1:1 (w/w, % VS basis) with an increased OLR of $3.5 \pm 0.5 \text{ g-VS}/(\text{L} \cdot \text{d})$ between days 55 and 76. After reaching the steady-state in both reactors on day 77, WF was gradually introduced in R1, replacing the same amount of VS of the DF in feed until only WF was used for co-digestion with FCM in feed of R1 (day 101). The feeding mixture in R2 was kept unchanged as on day 76 until day 214 when the experiments were terminated. From day 138 to day 180 FMM was added instead of FCM in both reactors.

Analytical methods

Analyses of TS, total suspended solids (TSS), total dissolved solids (TDS), volatile suspended solids (VSS), VS, and chemical oxygen demand (COD) were carried out for both raw and pretreated material. COD was determined in Hach Lange cuvette tests according to the company's method LCK 914. TS, TSS, VSS, and VS were analyzed in accordance with standard methods (American Public Health Association *et al.* 2005). Samples from both reactors were taken for measuring pH and VFAs two to three times per week. $125 \mu\text{L}$ of 17% H_3PO_4 was added in 1 mL of sample in a 2 mL Eppendorf tube and centrifuged at 14,000 rpm for 10 min. The supernatant was transferred into VFA vials for analysis in a gas chromatograph

PerkinElmer Clarus 400 series, equipped with flame ionization detector (FID) and a Hewlett Packard FFAP capillary column, $30 \text{ m} \times 0.53 \text{ mm}$ I.D., film thickness $1.0 \mu\text{m}$, using nitrogen as a carrier gas. The oven temperature was programmed from 115°C (hold for 3 min) to 125°C at a rate of $5^\circ\text{C}/\text{min}$ and then increasing $45^\circ\text{C}/\text{min}$ to 230°C and held at final temperature for 2 min. Nitrogen was used as a carrier gas at $18 \text{ mL}/\text{min}$ and the injector port and detector temperature were 175°C and 200°C respectively. Methane content (CH_4) in produced biogas for both batch and CSTR experiments was measured 2–3 times per week using GC (SRI-GC-310), SRI Instruments, USA, equipped with thermal conductivity detector and a packed column (Porapak-Q, $6 \text{ ft} \times 2.1 \text{ mm}$ I.D.), where nitrogen was also used as a carrier gas. The pH was measured using an InoLab® pH 727 meter (WTW Inc.), with precise measurement values (0.001 pH).

RESULTS AND DISCUSSION

Effect of wet explosion on substrate characteristics

The characteristics of DF and WEx-treated DF under five different treatment conditions (145-10, 165-10, 165-20, 165-10- O_2 , and 180-10) are displayed in Table 2. The TS and VS of the digested manure fibers were 20.0 and 14.8%, respectively, with a COD/VS ratio of 1.5. Generally, the COD/VS ratio did not alter significantly during WEx treatment (145-10, 165-10, 165-20 and 165-10- O_2). However, a significant higher COD/VS ratio of 1.7 was found for the WEx-treated fibers 180-10, where the treatment was performed at 180°C and 10 min treatment time. This may be explained by the fact that due to the higher temperature a higher amount of lignin was broken into lower molecular compounds like phenols with a higher COD/VS ratio.

Table 2 | Characteristics of digested manure fibers before and after WEx treatment under five different conditions

Batch	TS (g/L)	VS (g/L)	TSS % of TS	TDS % of TS	VSS (g/L)	COD (g/L)	COD/VS	pH
DF	199.5 (2.4)	147.6 (1.9)	89.4 (0.7)	10.6 (0.7)	136.1 (4.6)	218.5 (15.7)	1.48	8.31
145-10	120.1 (2.6)	89.5 (2.2)	86.5 (2.1)	13.5 (2.1)	78.3 (2.1)	135.2 (0.0)	1.51	9.04
165-10	121.0 (2.9)	89.9 (2.5)	82.8 (2.3)	17.2 (2.3)	75.7 (1.0)	131.3 (0.0)	1.46	8.79
165-20	118.6 (2.4)	89.6 (2.2)	83.9 (0.5)	16.1 (0.5)	74.3 (1.6)	130.8 (1.3)	1.46	8.84
165-10- O_2	109.7 (0.2)	80.7 (0.6)	77.9 (0.0)	22.1 (0.0)	53.5 (0.6)	123.2 (9.0)	1.53	7.60
180-10	120.5 (1.9)	89.9 (1.7)	80.0 (0.6)	20.0 (0.6)	71.9 (0.7)	152.7 (5.5)	1.70	8.80

Samples were diluted before WEx treatment (3:2); values in brackets are standard deviation.

The relatively high pH of the digested fibers even increased for all WEx treatment conditions except for batch 165-10-O₂ where O₂ was added. Furthermore, TS, and VS after the treatment 165-10-O₂ were lower than for the untreated material, obviously due to a higher conversion of the material to CO₂ by addition of O₂. The amount of TDS in the pretreated materials increased under all five conditions.

Change of methane yield by wet explosion

The course of methane production and the final methane yields during the batch digestion of DF and WEx-treated DF under the five tested WEx conditions are displayed in Figure 2. Generally, increasing the treatment temperature resulted in higher methane yields (Figure 2(a)). The highest methane yield (224 mL/g-VS) was found for the digested fibers treated at 165 °C under the addition of oxygen at a batch loading of 0.5 g-VS/vial, ISR 2 (Figure 2(b)).

At a higher load the final methane yield of the material treated with addition of O₂ was, however, significantly lower. This indicated an inhibiting effect from the WEx-treated material with oxygen that counteracted the increase in degradability. For all other treatment conditions the increase in loading of the batch vial had only a minor effect, indicating no or only low production of inhibiting compounds during WEx treatment. Without addition of oxygen, the WEx treatment at 180 °C for 10 min resulted in the highest increase of the methane yield of 136%.

CSTR Experiments

The performance of the test reactor R1 and control reactor R2 was monitored for 214 days by methane yield, VFA

concentration, and pH (Figure 3). In the initial start-up, both reactors were fed with FCM alone for 54 days. From day 55 untreated digested manure fibers were added to the feed of both reactors with the filtered manure. While maintaining the OLR at 3.5 ± 0.5 g-VS/(L · d) the methane yield per gram organic matter decreased in both reactors significantly from around 180 mL/g-VS_{added} to 118 and 111 mL/g-VS_{added}, in R1 and R2, respectively, due to the higher content of organic matter with a low degradability. After steady-state conditions were established in both reactors, the untreated fibers DF in the reactor feed of R1 were from day 77 gradually replaced by WEx-treated fibers WF, replacing 1/3 of the DF from day 76, 2/3 from day 90 and feeding 100% WF in co-digestion with FCM from day 101. The average methane yield in R1 increased in the following days gradually and reached on average 194 mL/g-VS_{added} (days 101 to 214) when feeding WF compared with 111 mL/g-VS_{added} in the control reactor R2 with untreated DF (Figure 3(a)). Both reactors show a decrease in methane yield from day 121 to 140 due to a process disturbance after blockage in the influent tube of both reactors, which made cleaning of the reactors necessary. The performance of both reactors recovered, however, during the period from day 137 to 180, when the fiber fractions were co-digested in both reactors with FMM. Despite these fluctuations the methane yield in R1 remained significantly higher than in R2 for all times.

During start-up of both reactors the VFA concentration in both reactors exhibited very similar patterns with a rise above 20 mM and subsequent decrease to values lower than 10 mM (Figure 3(b)). This indicated very similar performance of both reactors with an adaptation phase during start-up. Also when introducing WEx-treated fibers WF in R1 on day 76 the VFA concentrations remain

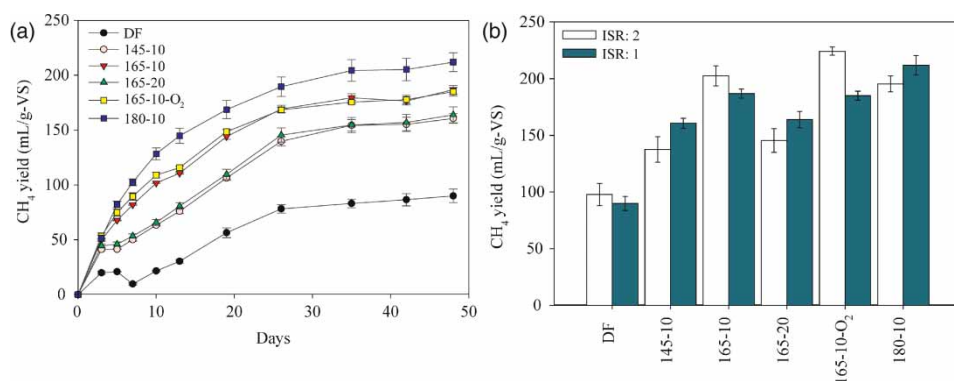


Figure 2 | Accumulated methane yield in batch experiments of DF and WEx-treated DF under five difference WEx conditions (145-10, 165-10, 165-20, 165-10-O₂, and 180-10). (a) Methane yields for a batch load of 1.0 g-VS/vial (ISR 1) and (b) final methane yields after 48 days for the two different batch loads (ISR 2 and ISR 1). Error bars indicate the standard deviation of three replications.

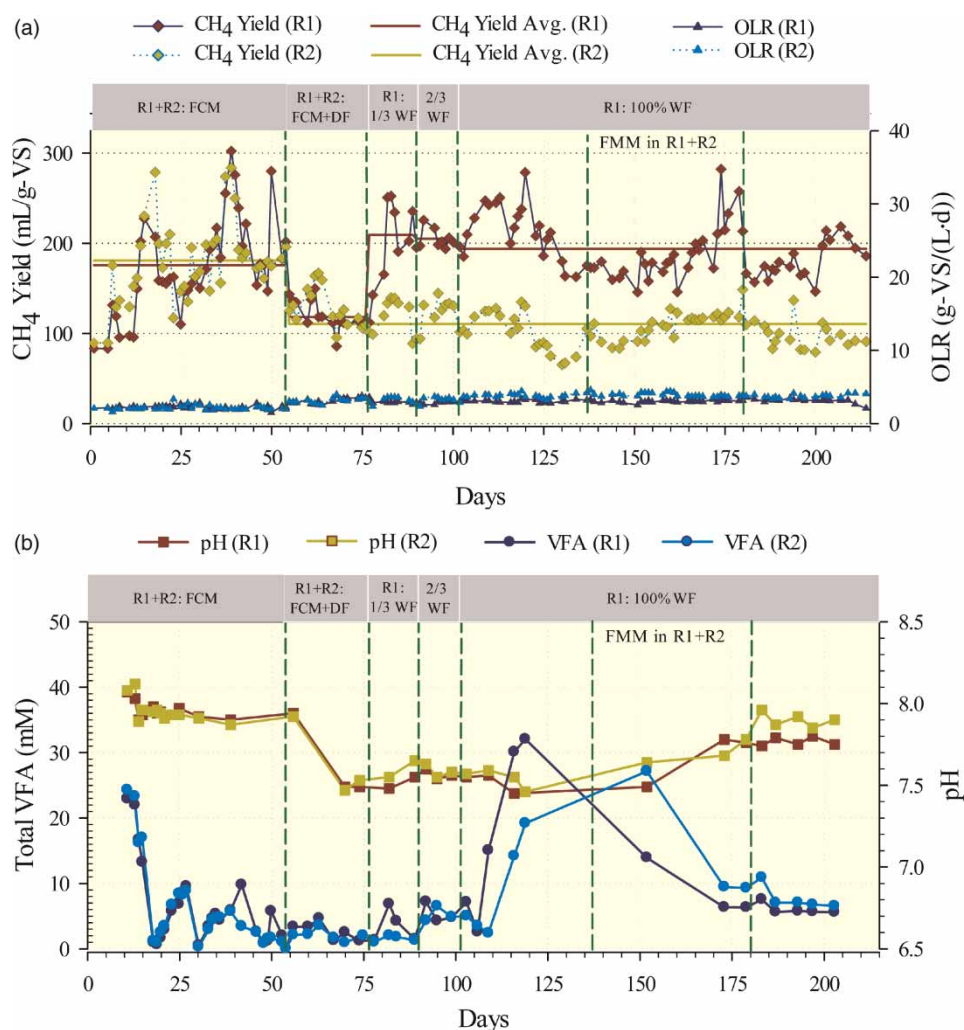


Figure 3 | Methane yield, OLR (a), VFA and pH (b) in reactor R1 and R2 in the different experimental phases. Phase 1 (until day 54): start-up of both reactors with FCM. Phase 2 (day 55–76): addition of digested fibers in R1 and R2. Phase 3–5 (day 77–214): change of feed in R1 to WF.

generally very similar in both reactors. In the period from day 107 to 119 a significant increase of VFA up to 19 mM in R2 and 32 mM in R1 was observed. Although the origin of this increase remained unclear the higher increase in R1 may indicate that the performance of reactor R1 was more sensitive when only WEx-treated fibers were added. On day 152 an increase of the VFA to 27 mM in R2 was caused by a process disturbance after blockage in the effluent tube. In the long run, however, and despite the change of FCM to FMM from day 137 to 180, the VFA concentration remained low in both R1 and R2. Furthermore, the generally very stable process performance of both reactors can be seen by the pH values of the reactors, which remained 7.6 ± 0.2 throughout the whole operation period.

Economy

The treatment of the digested fiber fraction in the new treatment concept offers two economical benefits: the biogas yield per ton of manure feedstock increases and the costs for treating only the separated digested fiber fraction are significantly lower than for the pretreatment of the whole reactor feed. In the case of Biokraft's biogas plant roughly 100 kg of separated digested fibers are leaving the reactor per ton of input. Consequently, the volume to be treated is only 10% compared with pretreatment of the whole input, reducing the operational costs accordingly. Through mass balance based on Biokraft's production data (Biokraft 2012) and the results found for increasing the biogas yield by WEx treatment of the digested fibers, it shows that the

methane yield per ton of feed can be increased from 23 to 28 m³ by the recirculation and to 33 m³ when the biodegradability of the fiber fraction is enhanced from 40 to 75%. This could make the new concept economically viable. More detailed values for investment and operational costs for the recirculation and WEx treatment will be available from a large-scale test.

CONCLUSION

The testing of a new concept for increasing the biogas yield of manure by combination of anaerobic digestion with wet explosion of the digested fiber fractions showed in both batch and reactor experiments that the methane yield of the fiber fraction can be significantly enhanced. Testing the WEx treatment under different conditions revealed optimum conditions at a temperature of 180 °C and a treatment time of 10 min without addition of oxygen, resulting in a 136% higher methane yield as compared with the untreated digested fibers in batch experiments. The continuous feeding of WEx-treated fibers in co-digestion with filtered manure revealed on average a 75% higher total yield. The batch experiments indicate that the addition of oxygen during the WEx treatment may lead to inhibiting compounds. The reactor experiments with digested fibers treated at 180 °C for 10 min revealed no significant signs of inhibition after a short adaptation phase when introducing WEx-treated fibers in co-digestion with manure. The proof of this concept in large scale will be followed by demo-scale testing of the concept at Biokraft's biogas plant on Bornholm.

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