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Continuous mesophilic anaerobic digestion of manure and rape oilcake – Experimental and modelling study

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ABSTRACT

Rape oilcake is a by-product formed after the removal of oil from rapeseed. Due to the high content of organic matter rape oilcake seems a good substrate for anaerobic digestion when it cannot be used as fodder. The aim of this work was to optimise the parameters used in a mathematical model of anaerobic digestion for rapeseed oilcake and cattle manure. The composition of these substrates was determined in order to estimate model inputs. Optimised kinetic constants of hydrolysis and decomposition for oilcake ($K_{\rm dis} = 0.77$, $K_{\rm hydCH} = 0.55$, $k_{\rm hydPr} = 0.57$, $k_{\rm hydlLi} = 0.30$) were estimated based on batch fermentation. The accuracy of the model with improved input parameters was confirmed by continuous fermentation. The average concentration of methane in biogas was about 50%. The biogas production efficiency from organic matter (defined as volatile solids) was 0.42 m³ kg⁻¹ with an organic substrate loading rate equal to 3.18 kg m⁻³ d⁻¹. The fermentation process demonstrated good stability and efficiency. The accuracy of the optimised model seems sufficient for use in modelling of a full scale process.

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1. Introduction

Rape (*Brassica napus*) is the main feedstock used for oil production in the European Union (the rapeseed harvest reached 20 Mt in 2009/2010) (Coyatte and Schenk, 2011). After the pressing process a by-product called oilcake accounts for 65% of seed mass. The majority of rape oilcake is used as fodder. About 4–9% of rape oilcake is not suitable for this application (because it is microbiologically contaminated during storage and transport) (Brzóska et al., 2010). Based on this value one can estimate that about 0.5–1.2 Mt of rapeseed oilcake in Europe currently remains unutilized annually.

Since the amount of organic matter in oilcakes is high, the use of this substrate for the production of renewable energy represents a potential solution. So far three possible ways of energy recovery from oilcakes have been reported: anaerobic digestion (Fernández-Cegrí et al., 2012), pyrolysis (Şen and Kar, 2011; Ucar and Ozkan, 2008) and combustion with fossil fuels.

Anaerobic digestion is a process in which the organic matter is converted to biogas (a mixture of methane, carbon dioxide and other trace compounds), biomass and mineral salts. Like the other two methods this method enables obtaining the energy

accumulated in the organic matter () but in addition it also allows the recovery of nitrogen and mineral salts. Furthermore the use of effluent as a fertilizer reduces the energy consumption required for the production of fertilizers (especially those containing nitrogen). The composition of oilcakes seems to make them appropriate

for use as a substrate for anaerobic digestion. The high mass fraction of relatively easily degradable polymers should ensure good biogas production efficiency. Several authors have reported the use of seed oilcakes as a substrate in anaerobic digestion (Bohdziewicz et al., 2012; Fernández-Cegrí et al., 2012; Staubmann et al., 1997). However the appropriate modelling of the process which is helpful during the introduction of new substrate on an industrial scale has not previously been presented.

An estimation of the possible impact of the addition of rapeseed oilcake to the other substrates traditionally used in biogas plants may be obtained from mathematical models such as the Anaerobic Digestion Model No. 1 (ADM1) (Batstone et al., 2002), developed by the International Water Association Task Group in 2002. To obtain a reliable simulation of the anaerobic digestion of a particular substrate some input variables and model parameters have to be optimised.

The parameters describing the composition of composite substrates (such as plant biomass) may be obtained using the approach presented by Koch et al. (2010). In this method the proportion of degradable biopolymers (proteins, carbohydrates and

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Nomenclature

TS1 total solids ADL acid detergent lignin VS volatile solids FOS/TAC volatile organic acids/total inorganic carbonate CP crude protein COD chemical oxygen demand crude lipids CI. NfF nitrogen free extracts CF crude fibre HRT hydraulic retention time **ADF** acid detergent fibre **NDF** neutral detergent fibre

lipids) in composite fraction are estimated on the basis of Weender analysis/van Soest extension (Naumann and Bassler, 1993; van Soest and Wine, 1967), and corrected by an appropriate factor to match the chemical oxygen demand (COD) unit used in ADM1.

The decomposition and hydrolysis of composite substrate is often a rate limiting step during anaerobic digestion (particularly in the case of plant material) (Vavilin et al., 2008), thus the kinetic rates of this reaction have a significant impact on the accuracy of simulation. Since the rate of disintegration and hydrolysis varies significantly depending on the substrates used in anaerobic digestion it is proposed to estimate the kinetic constant for individual substrates on the basis of batch experiments (Biernacki et al., 2013). However the batch test does not reproduce completely the fermentation conditions present in full scale installations and the model parameters obtained in this way should be compared with the results obtained from continuous fermentation (Deublein and Steinhauser, 2008).

The aim of this work was to investigate the possibility of using rape oilcakes as a substrate for anaerobic digestion and the determination of selected model inputs and parameters required for modelling with ADM1. To achieve this goal the composition of rapeseed oilcake was evaluated. In the second step the kinetic constants describing the disintegration and hydrolysis phases were optimised on the basis of a batch experiment, as proposed by Biernacki et al. (2013). In the last stage the accuracy of the parameters obtained was verified by the comparison of data from a continuous fermentation experiment with simulation.

2. Materials and methods

2.1. Substrates and inoculum

Rape plants (cultivar Sherlock and Digger) were cultured on a farm in Głubczyce – 50°12′N; 17°48′E (Poland). Seeds were harvested in July 2011. Rape oilcake was obtained by cold pressing in NAPUS-OIL S.C. – Kietrz 50°00′N; 18°00′E (Poland). The oil cake was stored at room temperature before use. The cow manure was obtained from a farm near Emden – 53°18′N; 7°12′E (Germany) in April 2012. For fermentation experiments solid particles were removed from the manure with a 3.6 mm sieve, to prevent the clogging of pumps and pipes connected to the reactor. Then the manure was mixed with an appropriate amount of water (in such a way that the manure mass was 50% of the total mass of substrate) and ground oilcake, and then stored at 4 °C until use.

As the inoculum the sludge from an anaerobic bioreactor was used. It was obtained from the agricultural biogas plant in Wittmund – 53°30′N; 7°54′E (EWE Biogas GmbH, Wittmund, Lower Saxony, Germany). This facility uses cow manure and food waste as a substrate. The inoculum was obtained in March 2012 and the following parameters were measured: pH, ammonium ion concentration, alkalinity, total volatile fatty acids, total solids and volatile solids (Table 1).

2.2. Batch experiments

The biogas batch experiments were prepared in accordance to VDI 4630 norm (Verein Deutscher Ingenieure, 2006), as it is described by Biernacki et al. (2013). The gas production from fermentation batch experiments was measured by the ANKOM Gas Production System (form ANKOM TECHNOLOGY) (ANKOM Technology, 2010). The following substrates were tested: rapeseed oilcake and sieved cow manure. Batch trials were prepared in 1.1 L borosilicate glass bottles. To 250 cm³ of inoculum 2.5 g of substrate was added, then the bottles were preincubated at 37 °C in a water bath for 1 h. After incubation the bottles were closed with ANKOM modules. The anaerobic digestion was wet tape performed under mesophilic conditions (temperature 37 °C). The sludge was manually mixed for 15 s every 24 h. All fermentation tests were prepared in triplicate. The border pressure in the system was set to 55 kPa. These experiments lasted for 21 days.

2.3. Continuous fermentation

For continuous fermentation a 25 L glass reactor with a water jacket was used. The temperature in the system was set to 37 °C and was maintained by a water bath with external circulation model E306 (Lauda Dr. R. Wobser GmbH & CO KG). The prepared substrate was stored at 4 °C and pumped into the reactor with a cavity pump model I-ID Type 0.03;10 (from Delasco PCM GmbH). A similar pump was used to remove the digested medium from the reactor. The fluid inside the reactor was continuously mixed at 0.833 Hz with a laboratory stirrer Eurostar power-B (from IKA - Werke GmbH & Co. KG). The production of biogas was measured with a gas counter model GT05/5 (from Dr.-Ing. Ritter Apparatebau GmbH & Co. KG) connected to a computer. The methane concentration (defined as a volume fraction) was measured with an UNOR 6 N infrared methane detector (SICK MAIHAK GmbH). Dosing pumps were turned on every 72 min. The volume of dosed substrate per day was about 0.66 L, which corresponded to a hydraulic retention time (HRT) of 30 days. The reactor was loaded with 15 L of inoculum and filled up to 20 L with manure; then after 2 days continuous feeding with manure was started. During the start-up period the reactor was fed with cow manure diluted with tap water (1:1) for 21 days. After this period rapeseed oilcake was added to the feeding substrate, while the concentration of cattle manure

Table 1 Inoculum parameters.

pН	8.2	
FOS/TAC	0.243	
Alkalinity (CaCO ₃)	9677	$ m mg~L^{-1}$
Org. acids (CH ₃ COOH)	2352	$ m mg~L^{-1}$
TS	4.08	%
Ash	1.47	%
VS	26.0	$\mathrm{g}\ \mathrm{L}^{-1}$
Ammonium – N	1440	$mg L^{-1}$

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