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Influence on anaerobic digestion by intermediate thermal hydrolysis of waste activated sludge and co-digested wheat straw

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This paper analyses time (30 and 60 min) and temperature (120–190 °C) effects of intermediate thermal hydrolysis (ITHP) in a two-step anaerobic digestion of waste activated sludge (WAS) with and without wheat straw as a co-substrate. Effects were analyzed by measuring biochemical methane potential for 60 days and assessing associated kinetic and chemical data. Compared to non-treatment, ITHP increased the secondary step methane yield from 52 to 222 L CH_4 kg VS^{-1} and from 147 to 224 L CH_4 kg VS^{-1} for pre-digested WAS and pre-co-digested WAS respectively at an optimum of 170 °C and 30 min. The hydrolysis coefficients (k_{hyd}) increased by up to 127% following treatment. Increasing ITHP time from 30 to 60 min showed ambiguous results regarding methane yields, whilst temperature had a clear and proportional effect on the concentrations of acetic acid. The energy balances were found to be poor and dewatering to increase total solids above the values tested here is necessary for this process to be energetically feasible.

1. Introduction

Pre-treatment of feedstocks is a common method to increase product yields in anaerobic digestion (AD) processes and has been the subject of several recent reviews, (Carlsson et al., 2012; Neumann et al., 2016; Pilli et al., 2014). Pre-treatment, as its name suggests, is usually performed *prior* to the process of AD. Several pre-treatment methods exist, e.g. chemical, physical, mechanical and biological, of which ultrasonic (mechanical) and thermal (physical) treatments are the main methods used regarding AD of wastewater treatment plant (WWTP) sludge (Carlsson et al., 2012).

The main reasons for pre-treating substrates prior to biogas production are to make recalcitrant organic matter more readily available to the microorganisms, consequently increasing CH_4 and CO_2 production kinetics, whilst reducing the final digestate volume and improving dewaterability. Thermal treatments are conducted at temperatures between 40 and 180 °C (Neyens & Baeyens, 2003), although this covers a wide range of effects from optimization of microbial activity at lower temperatures to high pressure operation at higher temperatures. The thermal hydrolysis process (THP) affects biodegradability, solubilization, viscosity, particle aggregations and dewaterability (Neyens & Baeyens, 2003; Oosterhuis et al., 2014) although a decrease in dewaterability due to THP has been reported at process temperatures below 160 °C (Takashima, 2008). THP of secondary sludge (waste activated sludge – WAS) can increase volatile solids (VS) reduction by over 60% compared to non-hydrolyzed WAS and digesters can be subjected to more than double the normal solids loading rate (Oosterhuis et al., 2014). WAS especially benefits from pre-treatment due to disintegration of its main characteristics, i.e. microbial flocks consisting of microorganisms and exopolymeric substances (EPS) (Carlsson et al., 2012). THP increases the available surface area, lyses cell walls where lipids are degraded to volatile fatty acids (VFAs) and cell wall associated carbohydrates are liberated.

As well as being used to treat biomass prior to AD, it is possible to apply THP as an intermediate step (ITHP), where a primary AD step is followed by THP after which the thermally hydrolyzed substrates are subjected to a second round of AD. The purpose of such

Abbreviations: AD, anaerobic digestion; ADp, preliminary anaerobic digestion; BMP, biochemical methane potential; B_0, ultimate methane yield; B_u, heoretical methane yield; EPS, extracellular polymeric substances; k_{hyd}, apparent hydrolysis constant; HPLC, high performance liquid chromatography; ITHP, intermediate thermal hydrolysis process; PCDS, pre-codigested waste activated sludge with straw; PDS, pre-digested waste activated sludge; ROA, rezex organic acid; STP, standard temperature and pressure; THP, thermal hydrolysis process; TS, total solids; VFA, volatile fatty acids; VS, volatile solids; WAS, waste activated sludge; WWTP, wastewater treatment plant.

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a process configuration is to further utilize the recalcitrant digestate's inherent energy. Remaining volatile solids content of sludge after AD is typically 60–70% of that prior to AD (Shana et al., 2013; Takashima, 2008) showing great potential to exploit it for further energy recovery. The advantage of ITHP over conventional THP is that the easily degradable components within a substrate will be biologically converted to biogas during the primary AD leaving only the more recalcitrant fractions for ITHP, thus saving some investment costs due to smaller scale equipment and operating costs resulting from the reduced amount of material to be treated. It has also been shown that ITHP improves biogas yield compared to THP (Shana et al., 2013). Available ITHP literature is limited and has mainly had its focus on specific substrate degradation kinetics and dewaterability rather than biogas production potentials as a consequence to different ITHP setups, i.e. temperature and time configurations (Shana et al., 2013; Takashima, 2008).

ITHP related research has usually been analyzing ITHP effects using flash, i.e. steam explosion, at the end of the THP process where the pressure is released quickly allowing the water to boil and thereby disintegrating fibrous materials (Pilli et al., 2014; Shana et al., 2013; Takashima, 2008). Steam explosion is a frequently used technology and has been examined previously, but not directly compared to THP without steam explosion (Dereix et al., 2006; Li et al., 2007). Besides the flash effect, there are other comparison issues when analyzing the THP effects due to differences in laboratory setups (BMP methods, volumetric v potentiometric gas measurements, inoculum composition and ratio etc.) and the actual character of the substrates, e.g. TS/VS, sludge sources, etc.

In addition to treatment of biomasses to improve methane yields, there is an increasing interest in the addition of wheat straw as a co-substrate in sludge AD to increase methane production at WWTPs. It has been estimated that world wheat straw production in 2010 stood at 846 million tons (Nges et al., 2015). It has a relatively large VS content and is an abundant and cheap substrate in certain geographical areas (Møller & Nielsen, 2016; Møller et al., 2004). Straw mainly consists of polymeric carbohydrates such as cellulose (glucose monomers) and hemicellulose (pentose monomers), which are the carbon source for methane production. However, for microorganisms to access this abundant energy source, they need to have the surrounding lignin structure altered or removed, as it acts as a strong barrier. Research into wheat straw co-digestion with manure and different pre-treatments has been published, for example Møller & Nielsen (2016), as have studies of co-digestion of primary sludge with wheat straw (Elsayed et al., 2016), of WAS with chicken manure and thermo-oxidatively treated wheat straw (Hassan et al., 2016) and of mixed sludges with wheat straw in a single digestion (Peng et al., 2016), but published work regarding the secondary digestion of pre-digested and pre-treated wheat straw with WAS has not come to the author's attention.

This paper analyses the aspects of ITHP on pre-digested WAS and pre-co-digested WAS with wheat straw in terms of methane production, production of monomeric carbohydrates and VFA at a variety of process temperatures and holding times of 30 or 60 min. The reasoning for using pre-digested WAS was to see the effects of ITHP on very recalcitrant material as was also seen in the work of Takashima (2008), who subjected the WAS to 120 °C for a duration of 60 min and found ITHP to be favorable with regards to VS destruction over both pre and post treatments.

2. Materials and methods

To test the efficiency of the thermal hydrolysis process at different temperatures and time of exposure and to test for possible advantages of straw addition, the setup depicted in Fig. 1 was arranged.

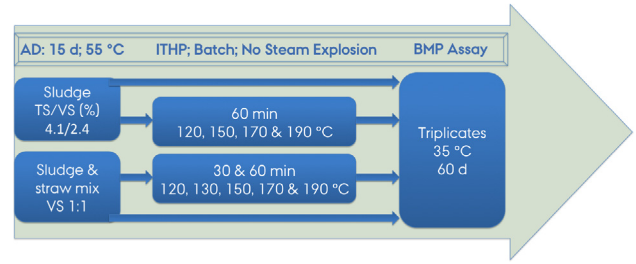

Fig. 1. Project flowchart.

2.1. Preliminary AD (AD_p)

Digesting the straw before the ITHP would simulate current interest in the co-digestion of agricultural waste products with wastewater sludges to increase methane yields at existing biogas reactors at wastewater treatment plants. Anaerobically digested WAS from Aalborg Vest WWTP, Denmark, was split into two sub-samples and wheat straw shredded to 10 mm particle size was added to one sub-sample. The second digestion was necessary as pre-digested WAS with straw was not available from a full-scale plant. The mix ratio of WAS to straw was 1:1 based on VS. The two different setups with pre-digested WAS and pre-co-digested WAS with straw addition will be referred to as "PDS" and "PCDS" respectively.

The AD_p for 15 days at 55 °C ± 1 °C was conducted in 20 L reactors with stirring velocity of 33 rpm, after which the digestate was immediately frozen in 1 L sealed polyurethane containers at −20 °C until the ITHP procedure. Total gas volume was measured with the "liquid displacement & buoyancy" system (www.bioprocess-control.com) with water acidified to ca. pH2 to prevent displacement of CO_2 (Valcke & Verstraete, 1983).

2.2. ITHP

The thermal hydrolysis treatments were performed in a high temperature and pressure bench top reactor (Parr 4524, www.parrinst.com) with a reactor volume of 2 L and electrical heating. Temperature and pressure data were monitored keeping the temperature range within ±1.5 °C of the target temperature. Electrical heating has been shown to make no difference in effect when compared to steam heating and therefore should be comparable to industrial methodology (Mottet et al., 2009).

To simulate industrial norm (~170 °C) and to compare with literature THP results, the temperature interval was chosen as 120–190 °C for 30 min and 60 min (Li & Noike, 1992; Neyens & Baeyens, 2003; Pilli et al., 2014; Pinnekamp, 1989; Shana et al., 2013; Takashima, 2008). To enable analyses of the ITHP effect a sample of each of PDS and PCDS were not subjected to ITHP and served as controls.

PDS samples were treated at 120 °C, 150 °C, 170 °C and 190 °C for 60 min, whilst PCDS samples were treated at 120 °C, 130 °C, 150 °C, 170 °C and 190 °C for 30 as well as 60 min to analyze time effects.

Samples were defrosted for 24 h at room temperature. Heating from 20 °C to target lasted 30–35 min, whilst cooling to <40 °C was accomplished in less than five minutes, after which the samples were frozen until the biochemical methane potential (BMP) assay. Freeze-thawing processes can act as a pre-treatment with large influences on dewaterability and cell lysis (Wang et al., 2001) but in this study all samples were frozen and results are thus comparable to each other.