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PILOT SCALE CONTINUOUS THERMAL HYDROLYSIS OF ORGANIC WASTES FOR INCREASED BIOGAS PRODUCTION

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Abstract

Thermal hydrolysis is an established method of pre-treating organic wastes prior to anaerobic digestion or as an intermediate step between two anaerobic digesters. The process can be likened to pressure cooking, where material is subjected to high temperatures (usually 100-200°C) and respective pressures for a defined period of time (usually <1 hour). For many full-scale and, to the best of our knowledge, all pilot and laboratory-scale applications, the equipment used usually operates in batch mode due to technical difficulties of adding and removing material to a pressurised continuous process. The advantages of a continuous (or semi-continuous) process at full-scale over a batch process include a smaller footprint and lower operational costs. At pilot or laboratory-scale, batch thermal hydrolysis reactors suffer from problems of slow heating and cooling times which leads to poor definition of true treatment times. This work describes the development and operation of a pilot scale (treatment volume of 1.02 litres) thermal hydrolysis system that operates semi continuously. By using a high electrical heating power of 7.2 kW and a high heating surface to volume ratio, the system can treat materials for periods of just a few minutes. The system has been tested using the liquid fraction of cattle manure and biogas batch tests have shown that methane yields were increased by up to 40.5% at four days digestion, but the improvement was less pronounced at longer digestion times, with 12% increased methane yield after thirty two days.

Keywords: Biogas, pre-treatment, thermal hydrolysis.
JEL Codes: Q40

Introduction

Pre-treatment to increase methane yields of organic wastes is becoming a very important subject following the growth of the anaerobic digestion (AD) industry and has been the subject of several literature reviews (Carlsson et al, 2012; Neumann et al, 2016). Pre-treatment can be achieved in many ways, such as mechanical, chemical or thermal methods, with the final goal of either increasing the long-term methane yield or increasing the rate of methane production so that a greater proportion of the yield can be achieved in a short timespan, thus allowing for smaller AD treatment plants.

Of the available pre-treatment methods, thermal treatment offers the advantage of lower maintenance costs than mechanical treatments and no materials costs as is the case with chemical treatments. The process requires a considerable thermal energy input but this is most often achieved by steam, produced by the burning of a portion of the produced biogas, and a considerable part of the energy input can be reclaimed through heat exchangers to transfer the energy to heating of the material flowing into the thermal hydrolysis reactor and to the AD reactors. It has been shown that thermal hydrolysis of waste activated sludge is energy self-sufficient in terms of heat requirement and biogas production (Chen et al, 2012).

Studies have been conducted on the thermal hydrolysis of animal manures (Raju et al, 2013; Budde et al, 2014), agricultural wastes such as corn stover (Darwin et al, 2016), food wastes (Yin et al, 2014) and for municipal sewage sludge with and without the addition of agricultural waste products (Bjerg-Nielsen et al, 2018). Animal manures and many agricultural wastes are lignocellulosic materials, thus the process must focus on the hydrolysis of plant fibres, whereas sewage sludge consists of microbial cells which require lysing prior to conversion to biogas. Thermal hydrolysis is also an accepted technology for the treatment of category 2 waste products as defined by the European Union (Directive 2002), which requires 20 minutes treatment at 133°C at a pressure of 3 bar, but if typically higher temperatures and treatment times are used in industry, even if the input material is not subject to the category 2 rules, this would suggest that energy is potentially being wasted. Although the goal of increased methane yields remains the same regardless of substrates, differences in the nature of substrates means there is a considerable degree of process optimisation required and thus the requirement for pilot and laboratory-scale equipment.

A serious disadvantage of smaller scale equipment suitable for thermal hydrolysis is that they usually operate in batch mode; they tend to consist of a pressure vessel with either electrical heating elements or steam injection. The robust nature of the pressure vessel means it has a considerable heat capacity and thus a lag time before the operating temperature is achieved (a problem which is realised in greater effect when electrical heating is used, based on the author's personal experience) and also a lag time to cool again to a safe temperature after the treatment time has been realised, which is true for both electrical and steam heated devices. There may also be problems with smaller scale equipment not being able to produce enough material for subsequent analysis in a single batch. At full-scale, rapid cooling is often achieved by the addition of dilution water to make the treated material suitable for pumping into a digester. However, this is unlikely to be practical at smaller scales.

This work describes the construction of a semi continuous thermal hydrolysis reactor at pilot-scale. The system uses a plug-flow reactor design with a two stage outlet and piston-controlled pressure release. The narrow

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tube reactor and high electrical heating power allow for very rapid heating of material whilst the pressure release piston and a cyclone cooler at the outlet allow for rapid cooling. Preliminary results using the liquid fraction of separated cattle manure as a substrate are presented in the form of biochemical methane potential (BMP) batch tests to determine ultimate methane yields.

Materials and methods

Reactor construction and operation

The construction of the reactor is shown in Figures 1 and 2. There follows a description of the construction where the various components are allocated a number that refers to the numbers annotated in Figures 1 and 2.

A stainless steel frame was constructed to mount the various components, complete with a step to allow for easy filling of the feed tank. The feed tank (1) has a capacity of ca. 70 litres and is stirred at 680 rpm by a propeller powered by a 250 W motor. From the feed tank the material is drawn into a progressive cavity pump (2) (Sydex, Lonigo-Vicenza, Italy) with a pressure differential of up to 24 bar. The hydrolysis reactor itself consists of 6 m of 14 mm internal diameter Inconel alloy tubing cut to four 1.5 m lengths (3) and connected with joining blocks (4) so that the direction of flow turns 180° three times to reduce the overall length of the device. The joining blocks between the lengths of tubing were constructed from 316 stainless steel machined to allow high-pressure tube fittings. The first joining block, just downstream of the pump, also contains a pressure sensor (5). Heating is by six “heat clamps” (6), each of which consists of two identical cast iron blocks with holes for a 600 W electric heating element in each block and a single PT100 temperature sensor located between the block pairs. The heat clamps are clamped around the Inconel tubing, with two runs of tubing through each block. Thus, the reactor had a total heating power of 7.2 kW which, in combination with the high surface area of the tubular reactor (surface area to volume ratio of 2.86 cm^2 per mL), allowed for rapid heating of the substrates. The PT100 sensors controlled the heaters via six proportional, integral, differential (PID) controllers. The system was designed with an upper temperature limit of 220°C although the software allows for temperature setpoints up to 999°C. The Inconel tubes, joining blocks and heat clamps were insulated with mineral wool. The exit chamber of the reactor consisted of a pneumatically operated DN25 ball valve (7) and a cyclinder of 50mm internal diameter (8) with a piston inside, all constructed from 316 stainless steel. The piston was sealed using hydraulic ram seals suitable for water-based materials, and was moved by a linear actuator (9) (Linak LA36, Nordborg, Denmark) with a thrust rating of 4800 Newtons. The final outlet of the reactor was a pneumatically operated DN50 ball valve (10) located between the previous ball valve and the cyclinder/piston assembly.

The principle of the reactor operation is that at a regular time interval, determined by the selected treatment time, the DN25 ball valve will open, allowing material under pressure to enter the cylinder (with piston extended towards the DN25 ball valve), then the DN25 valve will close, the piston will retract to a specific defined position, allowing a degree of depressurisation of the material in the cyclinder, before the DN50 valve is opened and the piston extends back to the previous position, with the treated material being ejected through the DN50 valve. The DN50 valve is then closed and the pump is started until the pressure in the reactor reached the value measured prior to the ejection cycle. The system then waits a period of time (again determined by the selected treatment time) before repeating the cycle. Downstream of the DN50 valve, the ejected material enters a 15 litre capacity cyclone cooler (11), where any remaining pressure is lost safely and the treated material is collected in a container placed beneath the cyclone.

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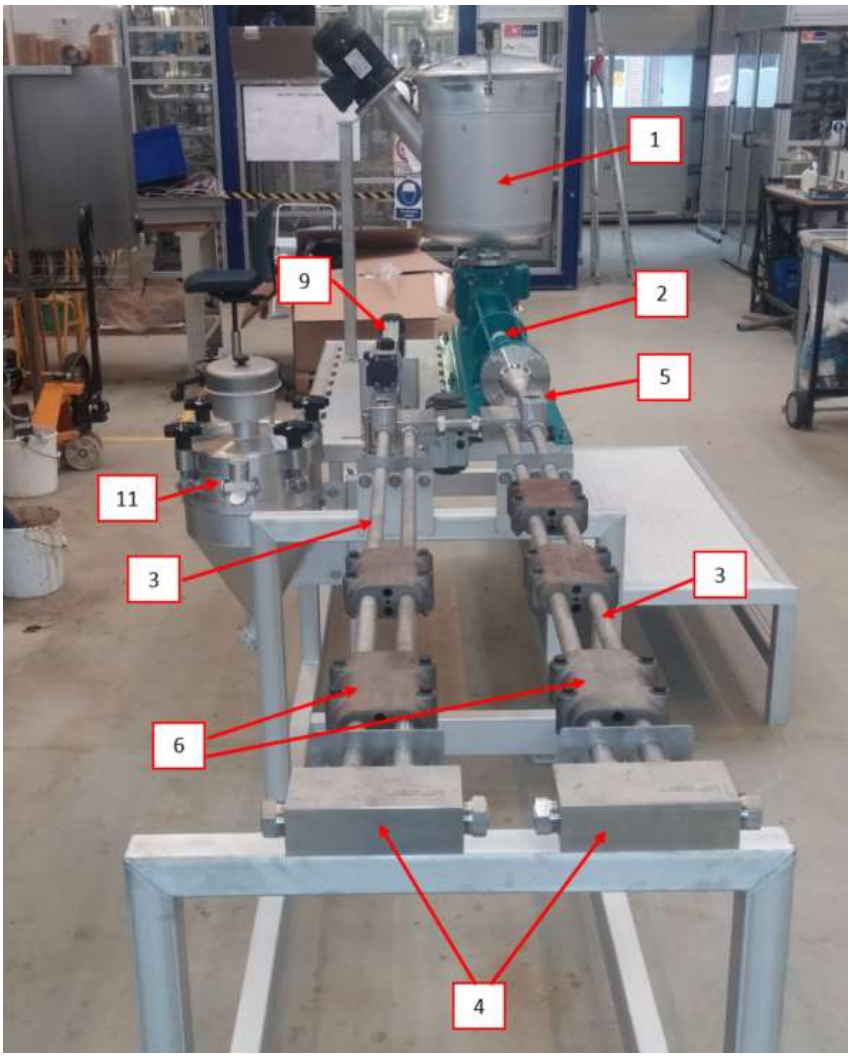

Figure 1. Overall view of the reactor (note that the electrical panel, mineral wool insulation and one of the heat clamps have been removed to give a clearer view)

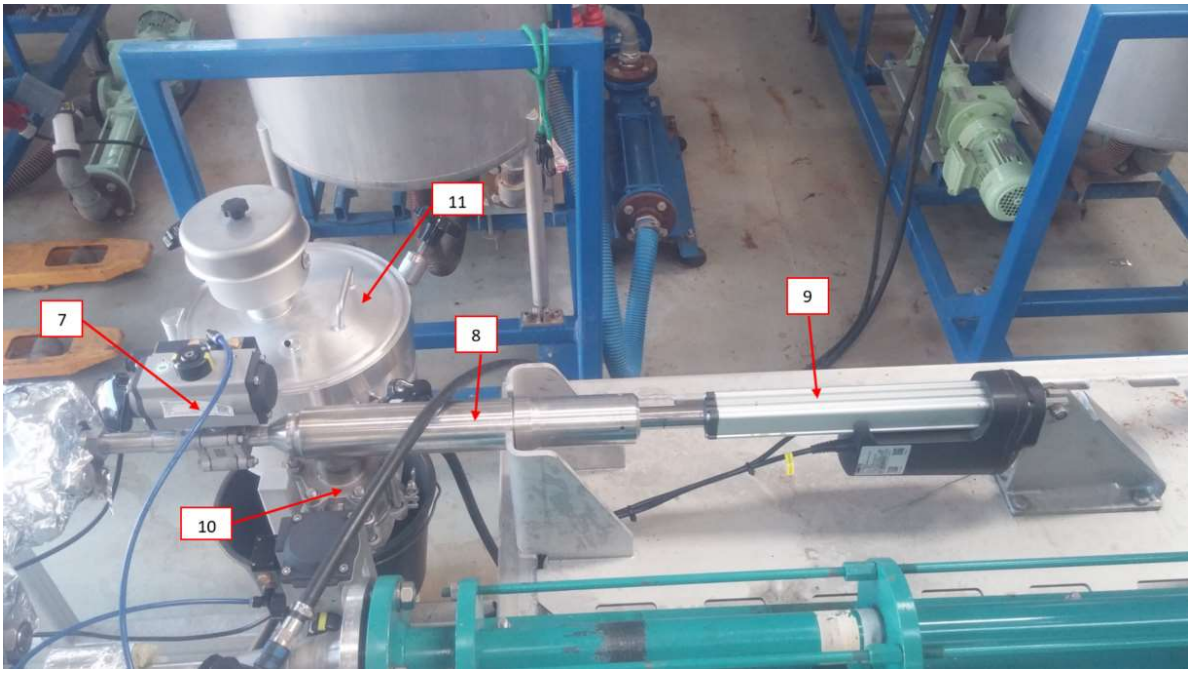

Figure 2. View of the exit chamber

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The reactor was tested for basic reliability and to check for leaks using water, then a more realistic test was performed using the liquid fraction of cattle manure that had been separated using a decanting centrifuge. This substrate was chosen because it was not expected to cause problems with blocking of the reactor tubes as it did not contain any fibres. For the preliminary tests, the reactor was operated at 150°C and at two treatment times of 10 and 20 minutes, hereafter referred to as $150^{(10)}$ and $150^{(20)}$, respectively, and the untreated material is hereafter referred to as UT.

BMP measurement

Dry matter (DM) and volatile solids (VS) were determined according to standard methods (APHA 2005).

The two treated samples and an untreated sample of the cattle manure were subjected to biogas batch assays to determine the ultimate methane yields, following the general procedure of Moset et al (2015). The batch assay was set up using 0.5 litre infusion bottles with butyl rubber stoppers. Assays were set up to a working volume of 250 mL. Each test was performed in triplicate, thus two treatments plus untreated plus control assays using inoculum alone, giving a total of twelve bottles. Microbially active inoculum was collected from a full-scale anaerobic digester (1100 m^3), treating ca. 63,000 kg d^{-1} of mixed cattle and pig manure with ca. 15,000 kg d^{-1} of straw, deep litter manure and grasses, thermophilically at 52°C with a hydraulic retention time of 15 days. The inoculum was filtered (1 mm) then temperature acclimated and de-gassed at 35°C for two weeks. The inoculum and substrate were mixed with a ratio of 2:1 in terms of VS. Each batch bottle was flushed with N_2 before incubation at 35°C for 32 days. Biogas production was measured using an acidified water displacement method as described by Feng et al (2017). The measured gas volumes were corrected to STP conditions and subtraction of water vapour pressure. Gas sub-samples were collected for biogas composition analysis at each measurement by gas chromatography (Agilent Technologies 7890A, CA 95051, USA) equipped with a thermal conductivity detector (TCD) and helium as the carrier gas. An Alltech CTR 1 double column (Grace, MD 21044, USA) was employed. The temperatures of oven, injector port, and detector were 120°C, 150°C and 150°C, respectively.

Results

Reactor operation

The reactor performed well during the preliminary tests described here. The heating sytem was tested with water and it was found that the reactor could maintain temperatures at treatment times as low as three minutes, although this is dependant on the operating temperature because there is a finite limit to how fast the incoming substrate can be heated. The upper limit of the treatment time is effectively unlimited although is restricted by the control system programming, which can be modified if necessary.

Substrate parameters

The substrate parameters are summarised in Table 1.

Table 1. Substrate parameters of untreated and treated materials

Material	DM (% w/w)	VS (% w/w)	pH
UT	4.16	3.18	8.23
$150^{(10)}$	4.11	3.08	7.48
$150^{(20)}$	4.15	3.13	7.46

Biogas yields

The cumulative biogas yields curves during the 32 day assay are shown in Figure 3. It is clear that $150^{(10)}$, with a peak value of 418 mL/gVS, produced more biogas than UT (peak value of 392 mL/gVS), yet $150^{(20)}$, with a peak value of 343 mL/gVS, produced less than UT at every measurement interval. This is further illustrated in Figure 4, which shows the percentage difference between the treatments and the untreated control. The greatest increase in biogas yield was for $150^{(10)}$ after only five days of digestion (15.4%) whereas the lowest was for $150^{(20)}$ at both 27 and 32 days (-12.4%). However, following Student's t-test analysis, the biogas yields were only significantly different from UT at days 5 and 7 for $150^{(10)}$ and at day 7 for $150^{(20)}$ due to variance between the replicates ($p<0.05$). The two treated sets were significantly different to each other at days 5 ($p<0.05$) and 7 ($p<0.01$), but not at longer digestion times.

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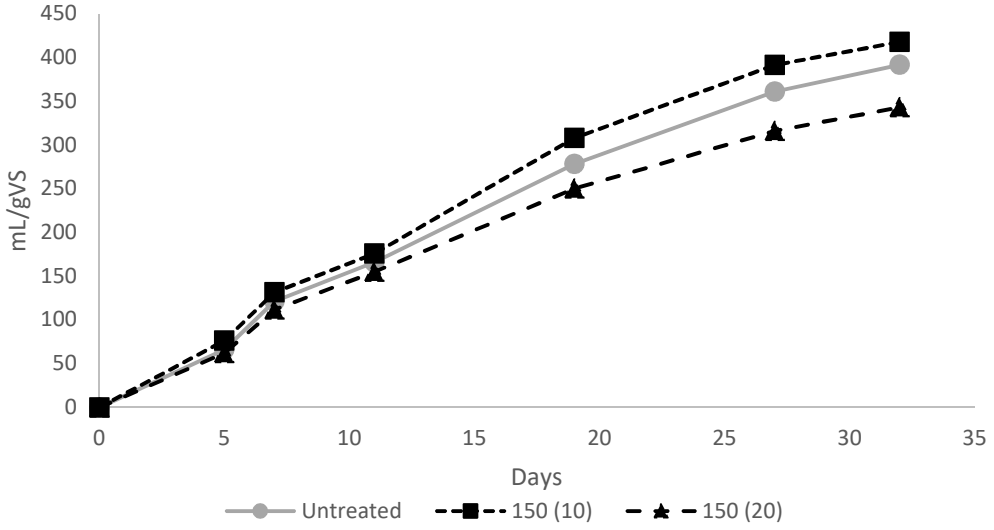

Figure 3. Cumulative biogas yields for the untreated substrates and substrates treated at 150°C for 10 and 20 minutes treatment

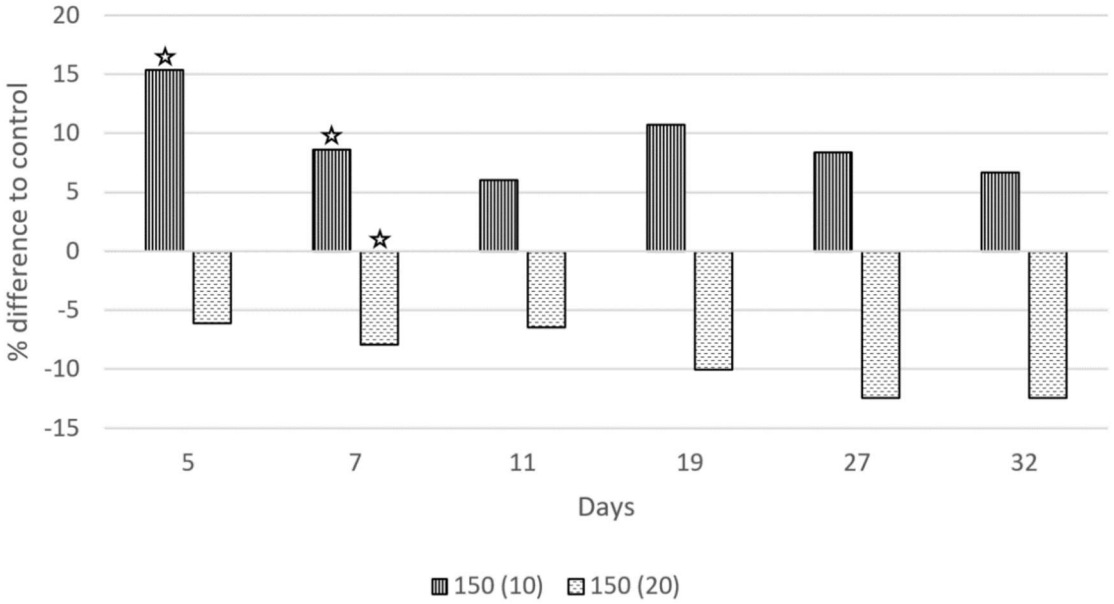

Figure 4. Percent difference in biogas yields of the two treatments compared to the untreated control. A single star above a bar indicates significance at p<0.05

Methane yields

The cumulative methane yields are shown in Figure 5 and the percent differences between treatments and UT are shown in Figure 6. The final yield achieved was 300 mL/gVS for $150^{(10)}$, whereas $150_{(20)}$ produced only 254 mL/gVS, with UT at 268 mL/gVS. For $150^{(10)}$, all measurement intervals showed significantly different methane yields to UT ($p<0.01$ on days 5 and 7, $p<0.05$ on all other days) whereas $150_{(20)}$ was only significantly different to the control at days 5 and 7 ($p<0.05$). Comparing $150^{(10)}$ and $150^{(20)}$ showed that the two sets were only significantly different on days 5 and 7 ($p<0.05$). The methane percentages at each measurement interval are shown in Figure 7 which shows that, except on day 19, $150^{(20)} > 150^{(10)} >$ UT and that this difference became less pronounced as the batch assay progressed.

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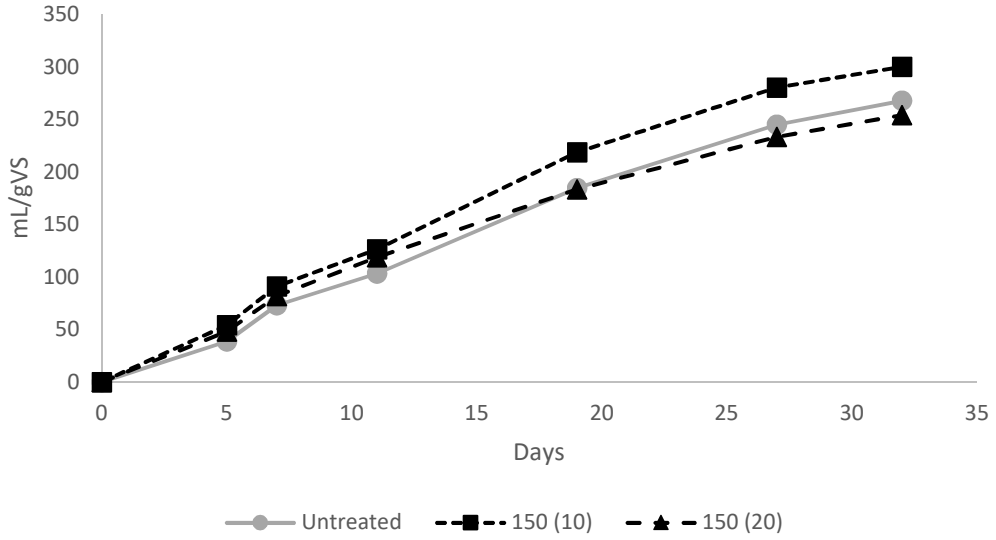

Figure 5. Cumulative methane yields for the untreated substrates and substrates treated at 150°C for 10 and 20 minutes treatment

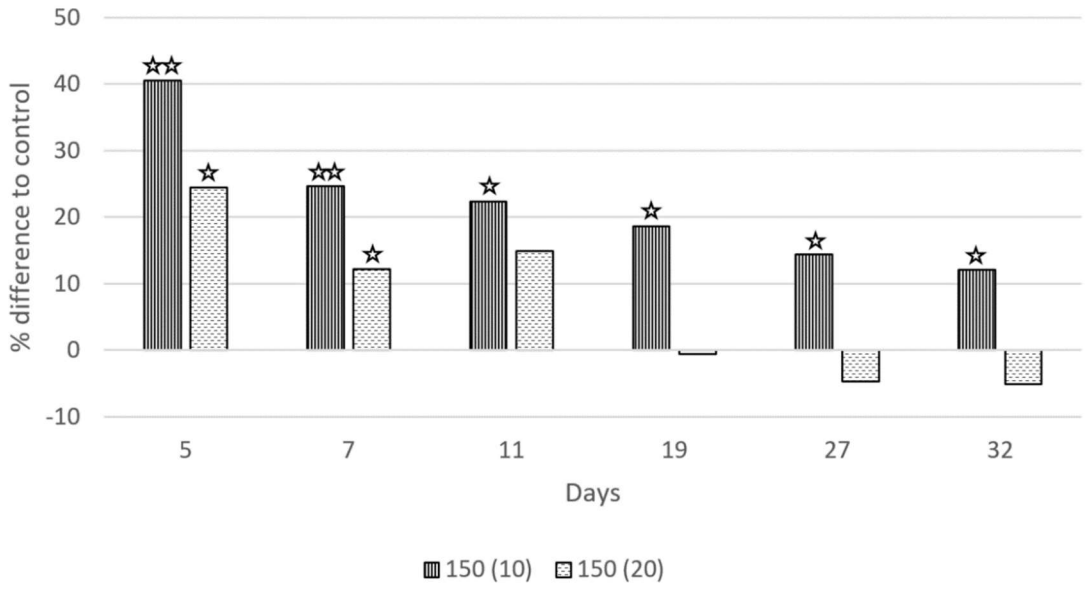

Figure 6. Percent difference in methane yields of the two treatments compared to the untreated control. A single star above a bar indicates significance at p<0.05, two stars indicate significance at p<0.01.

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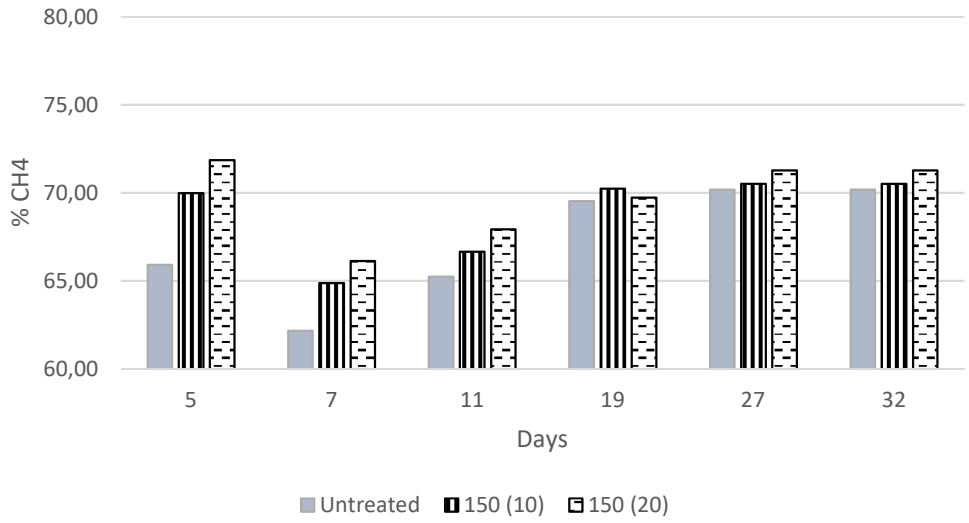

Figure 7. Methane percentages

Conclusions and discussion

The untreated manure methane yield of 268 mL/gVS is in agreement with Raju et al (2013) who published a yield of 281 mL/gVS for whole cattle manure and dos Anjos et al (2017) who measured 269 mL/gVS from the liquid fraction of cattle manure. The choice of the liquid fraction of separated manure as a substrate was not ideal for pre-treatment purposes, as treatments tend to focus on manure fibres (Raju et al, 2013; Kaparaju and Rintala, 2005) or sewage sludge (Bjerg-Nielsen et al, 2018) and the liquid fraction of cattle manure is lacking in the fibres present in the whole manure. However, the lack of particles removed the potential problem of the relatively narrow tubing becoming blocked, which was considered important during the first attempt with the new equipment. However, the tubing and associated joining blocks were identical to those used on a pilot-scale hydrothermal liquefaction reactor (Biller et al, 2018) which can handle biomass of 23% DM with the goal of producing bio-oil, where the pumping system (although different to that used in the reactor described here) is the limiting factor for the percentage DM. It is believed the thermal hydrolysis reactor can operate at DM values up to the maximum pumpable value, which for the progressive cavity type of pump is typically around 15% DM.

Raju et al (2013) reported a 21% increase in the methane yield of cattle manure following treatment at 200°C for 15 minutes, whereas an increase in pig manure methane yield of up to 29% was also found after 27 days of batch incubation, also for treatment at 200°C for 15 minutes. Huang et al (2017) published a 34% increase in methane yield of swine manure following treatment at 130°C for 30 minutes as well as an increased gas production rate, although higher temperature treatments were not attempted. Budde et al (2014) found a maximum of 58% increase in methane yield of cattle waste at 180°C for 5 minutes whereas at 220°C inhibitory compounds led to methane yields lower than the untreated control.

The greater increases in the yield of methane than that of biogas is attributed to the increased methane percentage in the biogas following treatment (Figure 7). The increased CH_4 concentration following treatment was attributed to a loss of CO_2 from the liquid phase at elevated temperatures. The reduced pH following treatment would also remove more CO_2 from the liquid. The pH of thermal hydrolysis treated material has been shown to have an inverse correlation to the treatment temperature, i.e. lower pH following higher temperature treatment, in a study of the thermal hydrolysis of food waste (Yin et al, 2014).

It is evident from Figures 3 and 5 that the cumulative gas yields had not reached a maximum, but the assay was stopped due to failure of replicates and therefore unsatisfactory standard deviations. This phenomenom only occurred in the treated samples, with the untreated replicates maintaining good repeatability. One of the three replicates for 10 minutes treatment suddenly experienced a cessation in gas production after day 32, whereas for the 20 minute treatment all three replicates deviated from day 19 onwards. It is not known why there was failure of treated replicates, but the single replicate of the $150^{(20)}$ set that maintained the highest gas yield produced similar results as the mean of the $150^{(10)}$ set. Therefore it cannot be claimed for certain that increased treatment time actually reduces yield, because the mean value was pulled down by poor repeatability, but it appears that treatment causes some sort of instability in subsequent batch assays, which may also be a cause of concern when treated substrate is added to continuous biogas reactors.

The behaviour of the material whilst inside the thermal hydrolysis reactor is unknown. The reactor design, as a relatively long and narrow tube, could show plug-flow characterstics but due to the low fluid velocity, laminar flow may be apparent, with the slowest fluid velocity closest to the tube walls and fastest velocity in the centre (Doran, 2013). A reduced fluid velocity close to the tube walls would lead to a broader residence time distribution and therefore imprecise treatment time. In addition, as the heating system is based on heat transfer from the heat

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blocks to the reactor tube and thence to the fluid, the fluid closest to the tube wall could experience higher temperatures than that at the centre. The reactor design attempts to increase fluid velocity by operating in a semi-continuous mode, with short pulses of higher fluid flow rather than a continuous lower flow rate. However, this also means the fluid is relatively static between pulses which could aggravate any temperature gradient problems. These uncertainties in the homogeneity of temperature can be problematic as higher temperatures can lead to problems; temperatures of 150-180°C can cause hemicellulose to hydrolyze and form inhibitory humic acids (Hendriks and Zeeman, 2009) and melanoidins (Stuckey and McCarty, 1984). Both treated substrates in this work appeared to have a darker brown colour which would suggest melanoidin formation, although this was not measured quantitatively. Similar observations were reported by Bjerg-Nielsen et al (2018). If the portion of the fluid closest to the reactor tube walls suffered higher temperatures and treatment times, and if the treated fluid was not completely homogenised, it is feasible that some batch assay bottles received a greater portion of inhibitory material than did others.

The thermal hydrolysis process can be described by the severity factor, using both treatment time and temperature (Alvira et al, 2010) and is written as in equation 1, where R_0 is the severity factor, t is time and T is temperature.

$$R_0 = t \times e^{[T-100/14.75]} \quad (1)$$

It is possible that the lower yields resulting from the 20 minute treatment time found in this work could be explained by greater severity (due to longer treatment time) leading to increased production of inhibitory substances. Bjerg-Nielsen et al (2018) also noted that thermal hydrolysis of sewage sludge with and without wheat straw was not significantly improved by increasing treatment time from 30 to 60 minutes.

It is important to remember that the gas yield results obtained here are from a batch test whereas the majority of full-scale processes operate on a semi-continuous basis, where substrate is added to a AD reactor regularly and a corresponding mass is also removed from the reactor to maintain the reactor mass. This usually leads to a stable biochemical environment, compared to the dynamic conditions in a batch test. The latter can be demonstrated by the non-linear cumulative gas yields in Figures 3 and 5. The best methane yield result of 40.5% greater than the untreated control was found at 5 days of batch digestion, which is a very short time compared to the mean retention time of continuous processes, which is typically at least 15 days. However, it is not possible to directly compare days of batch digestion with days of retention time in a continuous process. Nevertheless, an increased rate of gas production following treatment, which is demonstrated here by greater increases in yield at shorter digestion times, can be seen as an advantage in a continuous process.

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