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Ammonia removal in food waste anaerobic digestion using a side-stream stripping process

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Abstract
Three 35-L anaerobic digesters fed on source segregated food waste were coupled to side-stream ammonia stripping columns and operated semi-continuously over 300 days, with results in terms of performance and stability compared to those of a control digester without stripping. Biogas was used as the stripping medium, and the columns were operated under different conditions of temperature (55, 70, 85 ^{0}C), pH (unadjusted and pH 10), and RT (2 to 5 days). To reduce digester TAN concentrations to a useful level a high temperature ($\geq 70^0$C) and a pH of 10 were needed; under these conditions 48% of the TAN was removed over a 138-day period without any detrimental effects on digester performance. Other effects of the stripping process were an overall reduction in digestate organic nitrogen-containing fraction compared to the control and a recovery in the acetoclastic pathway when TAN concentration was 1770 ± 20 mg kg^{-1}.

Keywords: Ammonia removal; side-stream stripping; anaerobic digestion; food waste

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1 Introduction

The source segregation, separate collection and subsequent anaerobic digestion of food waste can help to reduce the organic fraction of municipal solid waste for disposal and, in some cases, help governments to meet the targets of the EU Directive on the landfilling of waste (1999/31/EC). Importantly, it also offers a method of reclaiming potential energy in the waste in the form of a fuel gas, and opens up a route by which nutrients can be recycled back to land. This has advantages even compared to incineration for energy recovery, as the high moisture content of food waste negates much of the energy gain and in thermal processing most nutrients are lost. Digestion may therefore offer a more sustainable route to resource recovery compared to other waste treatment technologies that are less suited to dealing with this high moisture fraction. Anaerobic digestion of food waste is not without difficulties, however, mainly associated with its high protein content. On hydrolysis this releases ammoniacal nitrogen which, although essential for the growth of anaerobic microorganisms, can lead to free ammonia concentrations that are inhibitory to the digestion process. The ammonia inhibits the methanogenic archaea, in particular the acetoclastic methanogens (Kayhanian, 1999, Chen et al., 2008, Liu and Sung, 2002, Prochazka et al., 2012, Angelidaki and Ahring, 1993). The result is operational instability, a decrease in biogas production, and in the worst cases failure of digestion. To some extent these problems have been resolved at mesophilic temperatures through stimulation of the hydrogenotrophic metabolic pathway by the addition of selenium and cobalt, both of which are commonly deficient in food waste (Climenhaga and Banks, 2008). This strategy has allowed stable digestion of food waste at high organic loading rates (OLR) (> 5 kg VS m^{-3} day^{-1}) and total ammoniacal nitrogen (TAN) concentrations > 6 g l^{-1} (Banks et al., 2012). At temperatures in the thermophilic range the toxic threshold is reduced as the equilibrium moves towards free ammonia, and under these conditions trace element additions have not been successful in overcoming the associated problem of volatile fatty acid (VFA) accumulation as the methanogenic/acetogenic syntrophy breaks down (Yirong et al., 2013). Yirong et al. (2013) compared mesophilic and thermophilic digestion of source segregated food waste without water addition into the system and found failure symptoms in the thermophilic system when TAN concentration reached 3.5 g N l^{-1}. To solve these operational problems in thermophilic anaerobic digestion of food waste one approach is to reduce the TAN concentration in the digester by dilution (Neiva Correia et al., 2008) but this has both resource and energy implications. Co-digestion to increase the C/N ratio is also possible, but depends on the availability of a suitable low nitrogen co-substrate. Reducing the ammonia in the digester or its feed are also possible solutions.

The application of ammonia stripping to the feedstock (pre-digestion) has been tested with piggery and poultry wastes (Zhang et al., 2011, Liao et al., 1995, Bonmati and Flotats, 2003, Gangagni Rao et al., 2008). Removal after first stage fermentation has been tested when treating abattoir, municipal and sewage sludge wastes (Resch et al., 2011, Nakashimada et al., 2008, Yabu et al., 2011). A side-stream process has been tested for slaughterhouse wastes after membrane separation at temperatures below 65 ^{o}C and pH 8.5 - 9 with NaOH addition (Siegrist et al., 2005); and for the liquid fraction of chicken manure digestate under 80 ^{o}C and a vacuum pressure of 600 mbar without pH adjustment (Belostotskiy et al., 2013). In both cases the free ammonia concentration

was maintained below the inhibition threshold. By using these techniques a wider range of high N feedstocks including food waste (domestic and commercial), abattoir waste and some animal manures are candidates for anaerobic digestion as a single substrate in both mesophilic and thermophilic conditions. The side-stream stripping process is particularly attractive as it is a simple 'bolt-on' concept that could be used with existing anaerobic digestion process designs. Additionally, nitrogen can be recovered as ammonium sulphate, an important nitrogen fertiliser source, and the use of nitrogen-reduced digestate allows a higher application rate in nitrogen-vulnerable zones under the Nitrates directive (91/676/EEC).

The aim of the application of side-stream stripping to anaerobic digesters treating food waste was to reduce the TAN concentration to a point where it would be unlikely to inhibit a thermophilic digester, analysing a number of different stripping conditions. It was also considered essential to monitor the digesters over an extended period to assess the long term effect of the stripping process, as the process itself subjects a portion of the digestate to both temperature and pH shock before returning it to the digester, with a potentially detrimental effect on digestion performance. Although the processes being developed in this research are primarily intended for use with thermophilic digestion the experiments used mesophilic conditions as the starting point since these allow operation at a high concentration of ammoniacal N in the digester, as necessary for demonstration of a side-stream process operating at a low bleed rate. The experiments were carried out against control digesters without side-stream interventions and in all cases a standard biogas of 65% CH_4 and 35% CO_2 (v/v) was used in the stripping process.

2 Material and methods

2.1 Digesters

Four 35-L working volume continuously-stirred tank reactors (CSTR) were used, constructed from PVC pipe sealed at its top and bottom with plates incorporating feed and drainage ports. Temperature was controlled by recirculating water from a thermostatic bath through an internal heating coil to keep the digesters at 36 ± 1 ^{o}C. The digesters were sealed from the outside atmosphere by a draught tube through which an offset bar stirrer was inserted to allow low speed mixing at 30 rpm by means of geared motors. Biogas production was measured using continuous gas flow meters (Walker et al., 2009). Gas yield was corrected to standard temperature and pressure (0 °C and 101.325 kPa). Biogas was also collected in a gas-impermeable bag for 1.5-hour periods starting 5 hours after reactor feeding; this sample was used to determine the biogas composition at least once per fortnight.

2.2 Digester inoculum

Inoculum was taken from digesters that had been acclimated to source segregated household food waste (OLR 2 g VS kg^{-1} day^{-1}) with trace element supplementation. These digesters had shown good performance and stability and had operated for over 4 hydraulic retention times (HRT) before the start of the current trial. The characteristics of the original inoculum used in the experiment are shown in Table 1.

2.3 Food waste

The digesters were fed on source segregated domestic food waste collected commercially by Veolia Environmental Services (UK), from homes in Eastleigh, UK. The waste was collected in biodegradable plastic bags and a representative sample of around 300 kg was taken periodically as required, from the same collection round. The food waste was taken out of the bags, and any obvious non-food contamination removed along with large bones and seeds. The sample was then ground (S52/010 Waste Disposer, IMC Limited, UK) to a homogeneous pulp, well mixed as a single batch and frozen at -18 °C in snap top plastic containers in ~4 kg aliquots. When needed, the feedstock was thawed and stored at 4 °C and used over a short period. The characteristics of the different batches of food waste used in the experiment are shown in Table 1.

2.4 Analytical methods

Total solids (TS) and volatile solids (VS) were measured according to Standard Method 2540 G (APHA, 2005) using an Heraeus Function Line Series oven and a 201/301 Carbolite muffle furnace. pH was determined using a Jenway 3010 meter (Bibby Scientific Ltd, UK) with a combination glass electrode calibrated in buffers at pH 4, 7 and 9.2 (Fisher Scientific, UK). Alkalinity was measured by titration with 0.25N H_2SO_4 to endpoints of pH 5.75 and 4.3 using an automatic digital titration burette system (SCHOTT titroline easy) to allow calculation of total (TA), partial (PA) and intermediate alkalinity (IA) (Ripley et al., 1986). Total Kjeldahl Nitrogen (TKN) indicates the sum of organic nitrogen (N_{org}) and TAN (ammonia and ammonium). TKN was determined after acid digestion by steam distillation and titration. This used a BÜCHI K-435 Digestion Unit with H_2SO_4 and K_2SO_4 as the reactants and $CuSO_4$ as the catalyst to convert the amino-nitrogen and free ammonia (NH_3) to ammonium (NH_4^+). This was then measured as TAN using a BÜCHI Distillation Unit K-350 with NaOH addition followed by collection of the distillate in boric acid indicator and titration with 0.25 N H_2SO_4. Volatile fatty acid concentrations (VFA) were determined by gas chromatography (Shimadzu GC-2010), with a flame ionization detector and a capillary column (SGE BP-21) and helium as carrier gas. Samples were acidified to 10% using formic acid and measured against mixed standards of 50, 250 and 500 mg l^{-1} of acetic, propionic, iso-butyric, n-butyric, iso-valeric, valeric, hexanoic and heptanoic acids (APHA, 2005). Biogas composition (CH_4 and CO_2) was determined using a Varian star 3400 CX Gas Chromatograph fitted with a packed stainless steel SUPELCO 80/100 mesh porapack-Q column and a TCD detector. The GC was calibrated with 65% CH_4 and 35% CO_2 (v/v).

The metabolic pathway for methanogenesis was determined by labelled [2-^{14}C] sodium acetate analysis on duplicate samples (Jiang, 2012). Each 15 g sample of digestate was mixed with anaerobic medium in the ratio of 1:2 and 0.15 ml of $^{14}CH_3COONa$ solution with a specific activity of 10 kBq ml^{-1} was added (MP biomedical, Solon, OH, USA). The mixture was incubated in 119 ml crimp top serum bottles at 37 ^oC for 48 hours. At the end of the incubation process the sample/medium mixture was acidified with 2 ml of 1mM H_2SO_4 and sparged using N_2 and O_2 gas mix (9:1 on a volume basis). The CO_2 and CH_4 produced were first passed through 20 ml 5M NaOH before CH_4 was oxidised to CO_2 in a tube furnace consisting of a heating block within which was embedded a quartz tube (6.2 mm OD, 4 mm ID, 180 mm length, H. Baumbach & Co Ltd, Suffolk, UK) packed with copper (II) oxide. The operating temperature was regulated at 800 ± 5

^{o}C using a temperature controller (Omega DP7004, Manchester, UK). The sparge gas then carried the CO_2 generated from CH_4 to a second CO_2 trap filled with 20 ml 1M NaOH. After absorption, 1 ml of each alkali trap and 1 ml of the centrifuged sample/medium mixture were added into 15 ml Gold Star multi-purpose liquid scintillation cocktail (Meridian Biotechnologies Ltd, Surry, UK) and counted in a Beckman Coulter LS6500 scintillation counter.

2.5 Ammonia stripping columns

Three of the digesters were coupled to stripping columns to remove ammonia in a semi-batch process. The stripping columns were made from stainless steel tube with a height of 56 cm and 10 cm internal diameter. Temperature was controlled using externally mounted thermostatically-controlled electrical heating mats (Non Adhesive Wire Wound Heater 104 Dia x 200 P 230V 200W; Holroyd, UK). Biogas was recirculated through the columns using a diaphragm pump (A.1F17N1.C06VDC; Parker, UK). The flow was adjusted using a rotameter set to a flow of 0.15 l min^{-1} $l^{-1}_{digestate}$ and the recirculated biogas entered the stripping column through a sintered-glass diffuser. The biogas leaving the column was passed through traps to remove ammonia: this was achieved by provision of a condensate trap followed by bubbling through deionised water and then through 0.25 N H_2SO_4 before recirculation to the stripping columns. The calibration of the rotameters was done by collecting biogas pumped over a fixed time in a gas-impermeable bag, then accurately measuring the volume using a weight gasometer (Walker et al., 2009). After each batch fill with digestate and replenishment of the ammonia traps the system was first flushed with biogas for 15 min to remove any air before switching to biogas. Figure 1a shows a schematic flow diagram of the biogas stripping apparatus.

2.6 Phase 1: Establishing a digestion baseline

After inoculation the digesters were initially operated for 122 days (1.14 HRT) at an OLR of 2 g VS kg^{-1} day^{-1} in order to establish a performance and stability baseline. Food waste feed was added daily and digestate was withdrawn twice a week. The digesters were operated with trace element supplementation following the recommendation of Banks et al. (2012) and monitored for pH, TAN, alkalinity, biogas production, gas composition, and volatile fatty acids.

2.7 Phase 2: Ammonia removal by side-stream stripping

A stripping column (or pair of stripping columns) was used in conjunction with a single digester and both the digester and stripping system were operated in semi-continuous mode. Feeding of the digesters and digestate removal continued as described in phase 1 but an additional portion of digestate, equivalent to 6% of the digester volume, was removed, sieved through a 1 mm mesh, and the liquor placed in the stripping column. The solids separated by sieving were immediately returned to the digester. After stripping for the required interval the treated liquor was returned to the digester from which it had been taken, with any volume loss compensated for by returning digestate from the wastage line. The conditions used in the stripping trials are detailed in Table 2. All the digesters were run with the same feedstock and at the same OLR irrespective of the operation of the side-stream stripping process.

During the course of the experiments a number of different stripping temperatures (55, 70 and 85 ^{o}C) were used in addition to pH control in some of the stripping tests. Where the pH in the stripping column was adjusted this was done by adding lime at 18.6 - 21.4 g CaO kg^{-1} of digestate (wet weight) to obtain a pH value around 10. One experiment also used two columns coupled to one reactor and operated independently. Success was measured in terms of TAN removal from the coupled digester as well as showing that no inhibition of the digestion process occurred as a result of the stripping process. A schematic diagram of the overall digester/stripping column coupled process is shown in Figure 1b.

3. Results and discussion

3.1 Phase 1: Baseline performance and stability assessment

All four digesters showed good performance over the first 122 days (1.14 HRT) despite having a high TAN concentration of 5.1 g N kg^{-1} and free ammonia around 500 mg N kg^{-1} (Figure 2). No VFA accumulation was detected (Figure 3a), the IA/PA ratio was less than 0.3 (Ripley et al., 1986), and VS destruction rates were 82.3, 83.6, 83.5 and 83.8 % in digesters 1 - 4 respectively. Specific biogas production was stable with values of 0.84 ± 0.03, 0.83 ± 0.03, 0.83 ± 0.04 and 0.82 ± 0.04 l g^{-1} VS (Figure 3b) and methane concentrations between 55-61 %.

Digestate characteristics are shown in Table 3. No noticeable upset was associated with the start-up of the digesters, but this was not surprising as the inoculum was taken from digesters that were being fed on the same substrate at the same OLR and had been receiving trace element supplementation.

3.2 Phase 2: Side-stream ammonia stripping

Side-stream stripping was coupled to the digesters between days 123 and 423, equal to 3 HRT based on food waste input and more than 4 retention times (RT) based on the internal HRT, i.e. taking into account the stripped digestate liquor returned to the digester.

The performance and the stability of the digesters did not appear to be affected by any of the measures introduced in the stripping columns. There was no major change in specific biogas production (Figure 3b) which remained stable during the side-stream stripping period (days 123-423). The measured methane concentration also remained steady at around 58 %. VFA concentrations remained below 400 mg l^{-1} (Figure 3a), although changes were seen in the alkalinity parameters (Figure 4) depending on the treatment imposed.

The purpose of the side-stream stripping was to reduce the TAN concentrations in the digesters, and the experiments tested the effectiveness of this under a number of different conditions. Changes in TAN are shown in Figure 2 for the different operational periods spanning days 123-423. Between days 123-260 the removal of TAN in digester R_1 coupled to the stripping column operated at 70 ^{o}C without pH adjustment was very similar to that in digester R_2, which was operated at the same temperature but with the pH adjusted to 10. Operation at a temperature of 55 ^{o}C and pH 10 gave a lower TAN removal apparently indicating that temperature was the main factor governing the

stripping process. During the first period (days 123-260) the stripping columns were operated with stripping gas connected to a biogas reservoir common to all the columns. During the second period (days 261-311) the stripping gas lines were separated, giving each column its own independent reservoir. As pH control had not appeared to be critical to TAN removal in the first period the addition of lime to the stripping column coupled to digester R_2 was also stopped. As temperature seemed to be the most important stripping criterion this was increased to 85 ^{o}C in the stripping column coupled to digester R_3. In an attempt to increase the rate of removal of TAN, a greater volume of digestate was removed from digester R_2 and loaded into two stripping columns working under the same conditions.

The results of these changes were surprising, in that digester TAN concentrations started to increase in R_1 and R_2, and there was no apparent improvement in the rate of TAN removal in R_3 despite the 30 ^{o}C increase in temperature. It was concluded that separating the gas stripping lines had caused this change, possibly due to preventing the precipitation of CO_2 and the enhancement of CH_4 content in the common stripping gas. In the previous experimental period this precipitation reaction resulted in a pH rise in the column without pH adjustment by lime addition, and this led to the incorrect conclusion that pH was of secondary significance compared to temperature. To demonstrate this, pH adjustment was reintroduced to one of the stripping columns coupled to digester R_2 on day 312. This resulted in an immediate reversal in the trend of TAN accumulation in the digester when compared to R_1, where stripping continued without pH adjustment but on an independent biogas recirculation loop. On day 326 pH adjustment was reintroduced in the stripping column coupled to digester R_1, and again a reversal in the trend of TAN in the digester was seen almost immediately (Figure 2).

On day 362 pH adjustment to the stripping column operating at 85 ^{o}C was introduced and the RT reduced to 2 days; this immediately increased the TAN removal rate to the highest level seen throughout the experimental trial.

Throughout the experimental period the control digester R_4 was run without side-stream stripping and this continued to show a TAN concentration in the digester > 5.0 g l^{-1}.

To determine the actual TAN removal in the stripping columns themselves, the TAN concentration was measured at the start and end of the stripping process for each of the stripping column conditions used. The results are shown as % TAN removal in Table 4. These confirm that both pH and temperature are important controlling factors and as both increase so does the % TAN removal, with the highest value achieved at 85 ^{o}C with pH 10.

To reduce the TAN to a point where it would be unlikely to inhibit a thermophilic food waste digester requires a concentration of ≤ 2500 mg l^{-1}. To achieve this in practice a side-stream striping process using both high temperature and pH adjustment would be necessary: this is borne out by the performance of digester R_3 which was coupled to a column operated at 55 ^{o}C and pH 10, but only showed an overall 21.0 % reduction in TAN compared to the control when operated over a 137-day period. Digester R_2, which had the longest operational period at high temperature and pH (128 days), showed an

overall TAN reduction of 48.2%, and the potential for even greater removal exists when using a higher temperature of 85 ^{o}C.

The use of side-stream stripping not only reduced digester TAN but also digester N_{org} content by between 20 - 33 % of the control value, with the greatest reduction corresponding to the high temperature and pH stripping conditions. This suggests that some additional hydrolysis is occurring as a result of temperature-mediated chemical processes. When the TAN removal profiles of the stripping columns are analysed in more detail (not shown) it can be seen that there is a clear 'lag' in TAN removal over the first several hours before the maximum rates of removal are observed. It is believed that this apparent lag is in fact due to further production of TAN in the stripping columns due to thermally-mediated alkaline hydrolysis of organic nitrogen-containing materials that have been carried over from the digester to the stripping columns. The ammonia released then contributes to the TAN removed in the column, and at the beginning of the batch stripping process the rate of TAN removal more or less equals the rate of fresh TAN production.

The bicarbonate alkalinity (PA) profile (Figure 4) shows a sharp increase due to the addition of lime. The CaO reacts with the CO_2 present in the bubbling biogas and precipitates as calcium carbonate. The alkalinity in digester R_1 between days 123 and 260 is lower than the control digester as NH_4^+ is also lost from the system. Figure 4 also shows that the IA in digester R_1 remained the same as in the control digester R_4 when there was no pH adjustment in the stripping column (days 123-260), whereas in digesters R_2 and R_3 it increased. An increase in the IA normally indicates a change in the concentration of VFA; however, this is not the case here as there is no indication of this occurring (Figure 3a). Increases in the IA/PA ratio show potential instability of the system and stable digesters typically have IA/PA ratios around 0.3 (Neiva Correia et al., 2008). During the baseline assessment (phase 1) IA/PA ratio fluctuated around 0.3. When coupled to the stripping columns the IA/PA ratio increased for all stripping conditions, but remained below 0.8 which is higher than the previous value but not uncommon in stable digesters with high alkalinity.

It is clear that the changes in alkalinity-related parameters are brought about by the conditions in the stripping columns, including the addition of lime to control pH which in turn removes CO_2 from solution. The removal of ammonia will also change the alkalinity and buffering capacity of the digesters. These changes did not, however, appear to effect the overall productivity of the system as measured by specific biogas production nor its stability as assessed directly by the concentration of VFA rather than by a change in the IA/PA ratio.

An increase in TS concentration was seen in digesters coupled to stripping columns in which the pH had been increased by the use of lime. This was the case between days 123 and 260 in digester R_2 and R_3 where the TS was 9.5-15.7 % higher than in the control (Figure 5). A similar observation was made from day 326 of operation for digesters R_1 and R_2 and from day 361 for R_3. A corresponding decrease in TS occurred between days 261-312 in digesters R_2 and R_3 when the pH in the stripping column was not increased: in both cases TS decreased until it reached that of the control digester. The TS concentration in digester R_1 between days 123-260 was 14.5 % lower than the

control. It is postulated that this may be due to the high temperature in the stripping reactor accelerating or improving the hydrolytic conversion. Evidence to support this comes from the observed slight decrease of VS in the reactors with side-stream stripping under all stripping conditions (Figure 5). It is thought that part of the VS of the liquor placed in the stripping columns is converted to VFA; in addition some of the N_{org} may also be broken down to ammoniacal nitrogen. This hypothesis also offers an explanation for the observation that there is no increase in TS and VS concentration over the duration of the stripping period, as might have been expected since water is lost from the stripping column as condensate. Without additional water production through improved hydrolysis both the TS and VS would be expected to rise.

Acetate oxidation activity is determined simply by measuring the production of $^{14}CH_4$ and $^{14}CO_2$ when labelled [2-^{14}C] sodium acetate is used in an incubation process. Labelled methane is exclusively formed when acetoclastic methanogens degrade acetate. In the syntrophic acetate oxidation pathway, however, both carbon atoms of acetate are converted to carbon dioxide and part of the carbon dioxide is consequently reduced to methane. Therefore, an increase in the $^{14}CO_2$:$^{14}CH_4$ ratio indicates a proliferation of the syntrophic acetate-oxidising pathway (Karakashev et al., 2006). Microbial ecology evaluated with fluorescent in situ hybridization and PCR temporal temperature gradient gel electrophoresis together with labelled [2-^{14}C] sodium acetate analysis conducted by Karakashev et al. (2006) on mesophilic and thermophilic full-scale digesters fed on manure and wastewater sewage sludge indicated that $^{14}CO_2$:$^{14}CH_4$ ratios below 0.1 were dominated by *Methanosaetaceae* and low levels of acetate oxidation, while $^{14}CO_2$:$^{14}CH_4$ ratio above 1 had high levels of acetate oxidation with populations dominated by other methanogenic Archaea and without *Methanosaetaceae*.

At the end of the experimental period the ^{14}C labelling assay showed an average $^{14}CO_2$:$^{14}CH_4$ ratio of 4.40 for the control reactor (TAN 5600 ± 70 mg kg^{-1}) (Table 5). This ratio indicates the dominant methanogenic pathway was via syntrophic acetate-oxidising bacteria. The same result was found by Jiang (2012), who detected a higher quantity of ^{14}C labelled carbon dioxide in the biogas when analysing food waste anaerobic digestate with high ammonia concentration (5-6 g N l^{-1}). The ratio in the ammonia-stripped digester R_2 (TAN 1770 ± 20 mg kg^{-1}) was 0.38, however, indicating that the acetoclastic route was now predominant in this case, even though the original inoculum for both digesters was the same and came from a digester in long-term operation on food waste. Schnurer and Nordberg (2008) showed a similar $^{14}CO_2$:$^{14}CH_4$ ratio between 0.5 - 0.8 for feedstock of diluted food waste with a low TAN concentration (0.65 - 0.9 g N l^{-1}), indicating that the main methanogenic pathway was acetoclastic. They also supplemented a reactor with egg albumin to increase the TAN concentration, and found that at 5.5 g N l^{-1} the mechanism had clearly shifted to syntrophic acetate oxidation ($^{14}CO_2$:$^{14}CH_4$ ratio above 2). Therefore, the current result confirms that even after long-term operation on food waste (123 days without stripping and 300 days with side-stream stripping) the acetoclastic population can be recovered when TAN concentration is decreased by side-stream stripping.

The experiments showed that side-stream stripping was effective in reducing the total ammonia nitrogen in mesophilic food waste digestate, starting from a relatively high concentration that would have been toxic under thermophilic conditions. Removal of a

proportion of the digester contents and exposure of them to thermophilic conditions with pH adjustment had no adverse effect on performance in terms of gas production or VS destruction. The research thus shows the way forward to the application of this technique in preventing the build-up of ammonia in thermophilic conditions, if the digester is initially set up with a low-nitrogen inoculum. The potential to control the nitrogen content also opens up the possibility of creating 'designer digestates' in which the balance of nutrients is tailored to the soil type and crop needs; while the extracted ammonia is itself a valuable fertiliser product for application during crop growth (Gowariker et al., 2009).

4 Conclusions

Side-stream stripping of ammonia using thermal alkaline treatment was effective and had no adverse effect on performance or stability of the digestion process at the bleed rate used in these experiments. The process required high pH and temperature to achieve a TAN concentration below the toxic threshold for thermophilic digestion, and it is unlikely that stripping at 55 ^{o}C and pH 10 would achieve the target reduction. This could, however, be achieved at ≥70 ^{o}C. The use of side-stream stripping not only reduced TAN but also N_{org}, possibly due to additional temperature-mediated alkaline hydrolysis in the stripping column.

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Table 1 Inoculum and feedstock characteristics

Inoculum characteristics

		Average	Deviation	max	min
pH		7.9	0.01	7.91	7.89
TA g l^{-1}	g l^{-1}	23.9	0.4	24.2	23.6
PA g l^{-1}	g l^{-1}	18	0.5	18.4	17.6
IA g l^{-1}	g l^{-1}	5.2	0	5.2	5.2
TAN	g kg^{-1}	4.86	0.07	4.91	4.81
TKN	g kg^{-1}	8.75	0.04	8.78	8.72
TS	g kg^{-1}	66.3	0.6	66.7	65.8
VS	g kg^{-1}	48.3	0.4	48.5	48
VFA	mg l^{-1}	148	6	152	143

Characteristics of the food waste batches

N	Start (feeding day)	End (feeding day)	TS (g kg^{-1})	VS (g kg^{-1})	TKN (% dry)
1	0	55	246.2 ± 2.4	228.1 ± 1.4	-
2	56	70	232.7 ± 3.8	211.8 ± 1.8	-
3	71	162	218.6 ± 6.2	202.9 ± 5.9	3.7 ± 0.5
4	163	227	209.8 ± 0.9	183.4 ± 0.3	3.6 ± 0.1
5	228	270	218.6 ± 6.2	202.9 ± 5.9	3.7 ± 0.5
6	271	334	239.8 ± 4.7	218.2 ± 4.3	3.5 ± 0.1
7	335	403	229.3 ± 1.2	208.1 ± 2.4	3.01 ± 0.04
8	404	423	249.1 ± 3.9	232.2 ± 3.8	3.2 ± 0.1

TKN (g N kg^{-1}) = TKN (% dry) x TS(g kg^{-1}) :100

Table 2 Conditions used in side-stream stripping experiments

	Days 123 - 260	Days 261 - 311	Days 312 - 325	Days 326 - 361	Days 361 - 423
R_1	C_1 T: 70 °C, pH n/a, RT: 4 day; SP: 1.5% day^{-1}	as before	as before	C_1 T: 70 °C, pH 10, RT: 3 day; SP: 2% day^{-1}	as before
R_2	C_2 T: 70 °C, pH 10, RT: 3 day; SP: 2% day^{-1}	C_2 T: 70 °C, pH n/a, RT: 4 day; C_4 T: 70 °C, pH n/a, RT: 4 day; SP: 3% day^{-1}	C_2 T: 70 °C, pH 10, RT: 3 day; C_4 T: 70 °C, pH n/a, RT: 4 day; SP: 3.5% day^{-1}	as before	as before
R_3	C_3 T: 55 °C, pH 10, RT: 5 day; SP: 1.2% day^{-1}	C_3 T: 85 °C, pH n/a, RT: 3 day; SP: 2% day^{-1}	as before	as before	C_3 T: 85 °C, pH 10, RT: 2 day; SP: 3% day^{-1}
R_4	Control, no stripping column				

R_1 - R_4 = anaerobic reactor 1 to 4; C_1 - C_4 = stripping column 1 to 4; T = temperature; RT = retention time; SP = reactor portion stripped per day; n/a = not adjusted

Table 3 Digestate characteristics without side-stream stripping (average day 0 to 122)

		R_1	R_2	R_3	R_4
pH		7.98 ± 0.07	7.96 ± 0.06	7.94 ± 0.07	7.93 ± 0.06
TA	g l^{-1}	25.1 ± 0.9	25.0 ± 1.0	24.8 ± 0.9	25.0 ± 1.1
PA	g l^{-1}	18.6 ± 0.8	18.0 ± 1.0	18.4 ± 0.7	18.9 ± 0.9
IA	g l^{-1}	5.8 ± 0.4	5.7 ± 0.6	5.7 ± 0.5	5.3 ± 0.8
TAN	g kg^{-1}	5.1 ± 0.01	5.1 ± 0.01	5.1 ± 0.01	5.1 ± 0.01
TKN	g kg^{-1}	8.75 ± 0.04	8.75 ± 0.04	8.75 ± 0.04	8.75 ± 0.04
TS	g kg^{-1}	64.5 ± 1.1	64.4 ± 1.4	65.5 ± 1.9	64.3 ± 0.9
VS	g kg^{-1}	47.4 ± 0.6	47.4 ± 1.0	47.9 ± 1.1	47.1 ± 0.6
VFA	mg l^{-1}	270 ± 100	260 ± 100	270 ± 80	290 ± 120

Table 4 TAN concentration (average) decreased per day

	% TAN decrease day $^{-1}$
55 ^{0}C pH 10	6.8
70 ^{0}C unadjusted pH	15.4
70 ^{0}C pH 10	21.1
85 ^{0}C unadjusted pH	16.4
85 ^{0}C pH 10	32.4

Table 5 Results from ^{14}C labelling experiment

Sample		**^{14}C** kBq	**Count Eff.[a]**	**Total ^{14}C recovered** kBq	**% Rec[b]**	**$^{14}CO_2$:$^{14}CH_4$**	**TAN (FA)** mg kg^{-1}
R_2 1	Sludge	0.67	84.11				
	CO_2	0.22	95.19	1.40	93%	**0.42**	
	CH_4	0.51	95.16				1770 ± 20
R_2 2	Sludge	0.69	83.21				(99 ± 1)
	CO_2	0.17	95.18	1.37	91%	**0.34**	
	CH_4	0.51	95.15				
R_4 (control) 1	Sludge	1.00	87.59				
	CO_2	0.26	94.92	1.31	87%	**4.43**	
	CH_4	0.06	95.05				5600 ± 70
R_4 (control) 2	Sludge	1.00	87.57				(500 ± 6)
	CO_2	0.27	95.07	1.32	88%	**4.37**	
	CH_4	0.06	95.15				

[a] Counting efficiency determined by scintillation counting

[b] Recovery rate including kBq recovered from sample/medium mixture, 5M NaOH trap and 1M NaOH trap. 1.50 kBq was the initial dose in the anaerobic medium.

Figure 1.

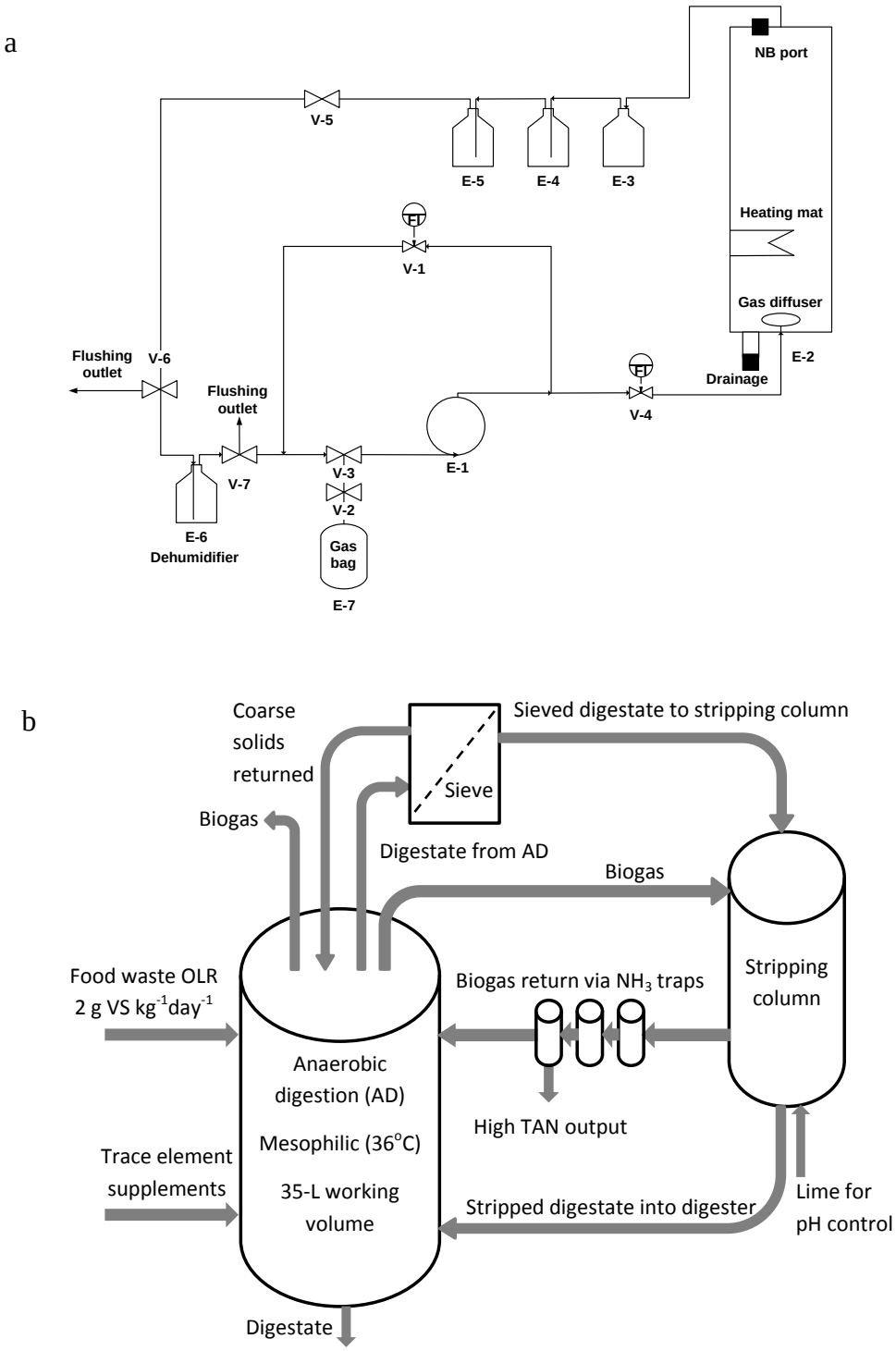

Figure 1. Details of experimental set-up (a) Process flow diagram for stripping column. E-1 diaphragm pump, E-2 stripping column, E-3 condensate trap, E-4 water trap, E-5 0.25N H_2SO_4 trap, E-6 dehumidifier, E-7 gas bag; (b) Schematic of the coupled process.

Figure 2.

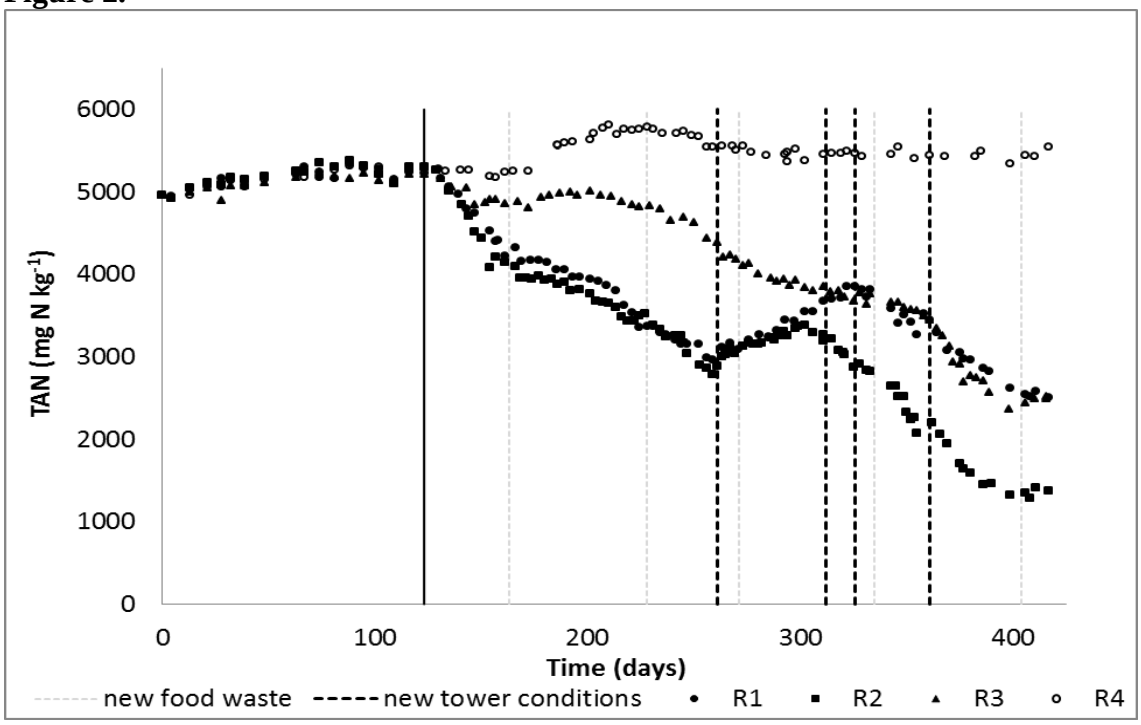

Figure 2. Total ammonia nitrogen in digestate during the experimental period. R_1: closed circle. R_2: closed square. R_3: closed triangle. R_4: open circle. Black continuous vertical line indicates start of stripping. Black discontinuous vertical lines indicate a change in stripping conditions (Table 2). Grey discontinuous vertical lines indicate a new batch of food waste (Table 1).

Figure 3.

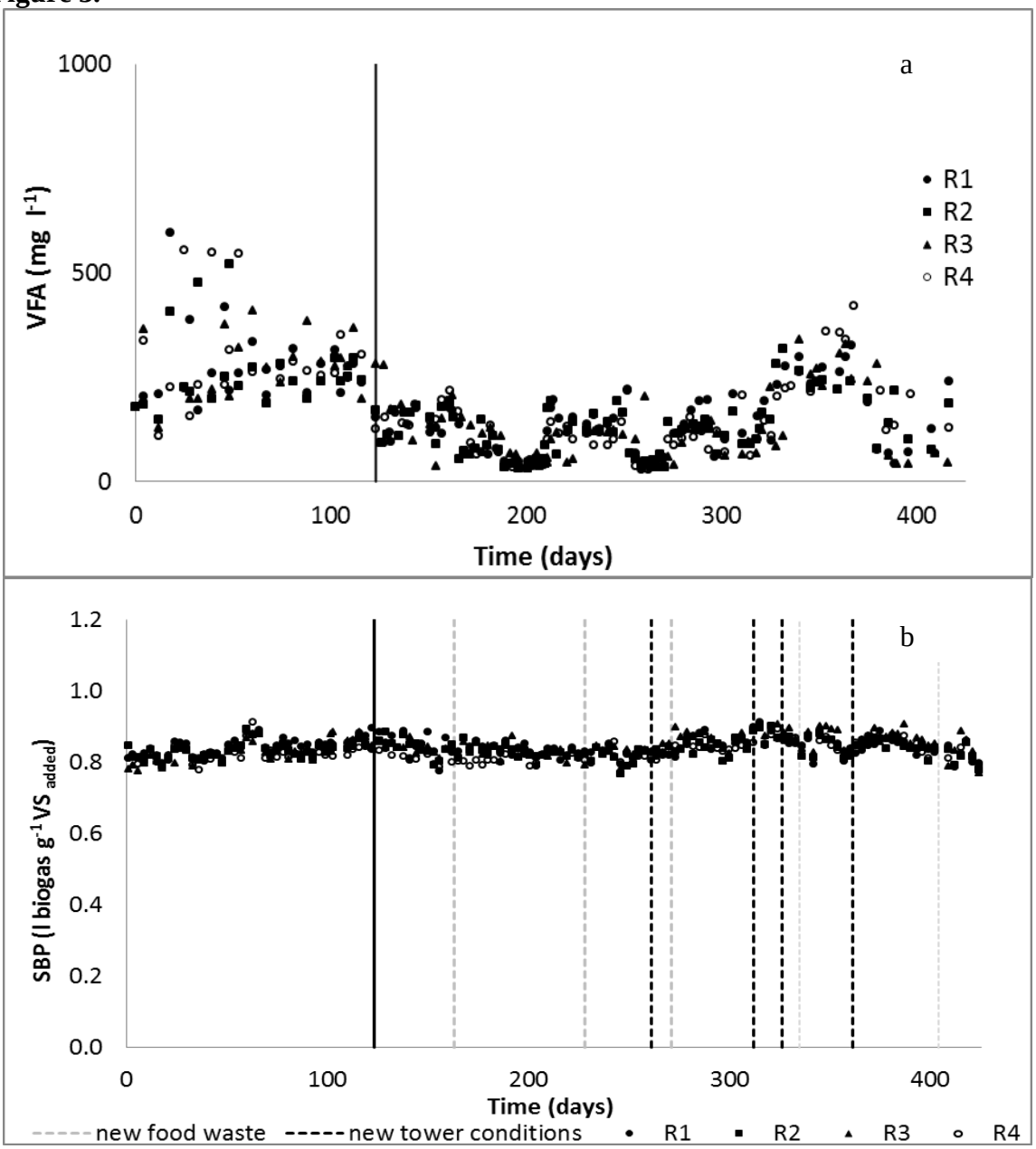

Figure 3. Total volatile fatty acid concentrations (a) and specific biogas production (b) during the experimental period. R_1: closed circle. R_2: closed square. R_3: closed triangle. R_4: open circle. Black continuous vertical line indicates start of stripping. Black discontinuous vertical lines indicate a change in stripping conditions (Table 2). Grey discontinuous vertical lines indicate a new batch of food waste (Table 1).

Figure 4.

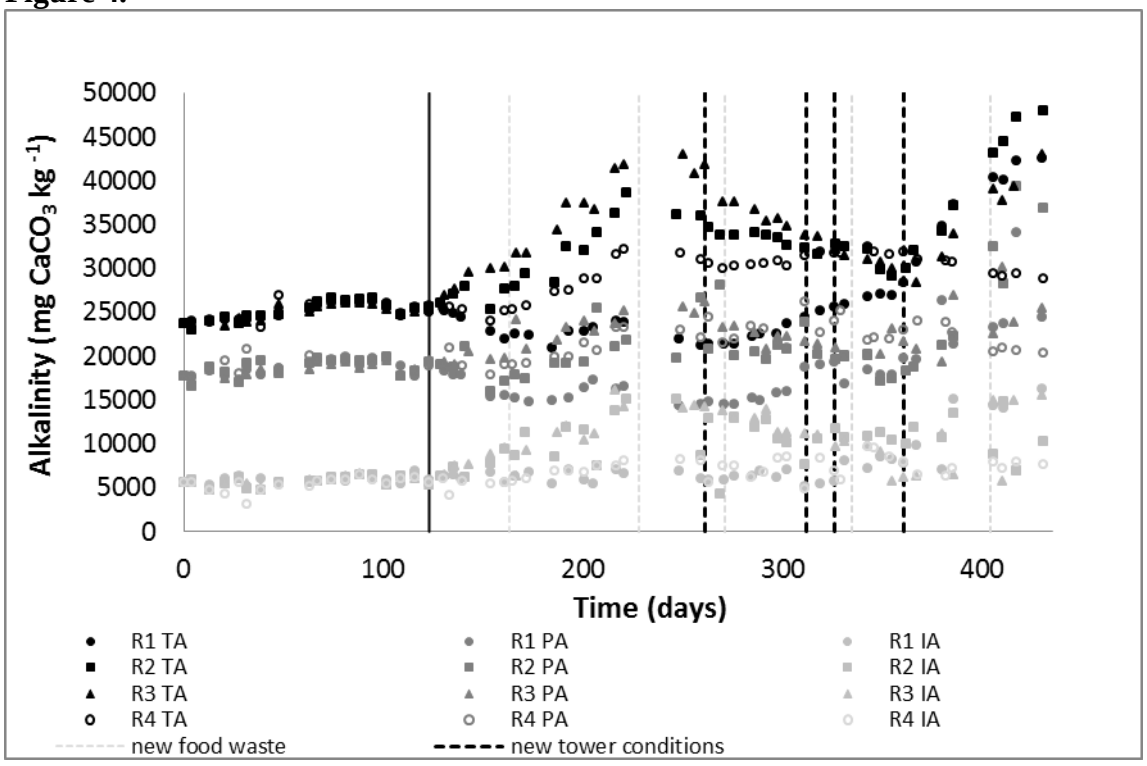

Figure 4. TA, PA and IA of digestate during the experimental period (black: TA, grey: PA, light-grey: IA). R_1: closed circle. R_2: closed square. R_3: closed triangle. R_4: open circle. Black continuous vertical line indicates start of stripping. Black discontinuous vertical lines indicate a change in stripping conditions (Table 2). Grey discontinuous vertical lines indicate a new batch of food waste (Table 1).

Figure 5.

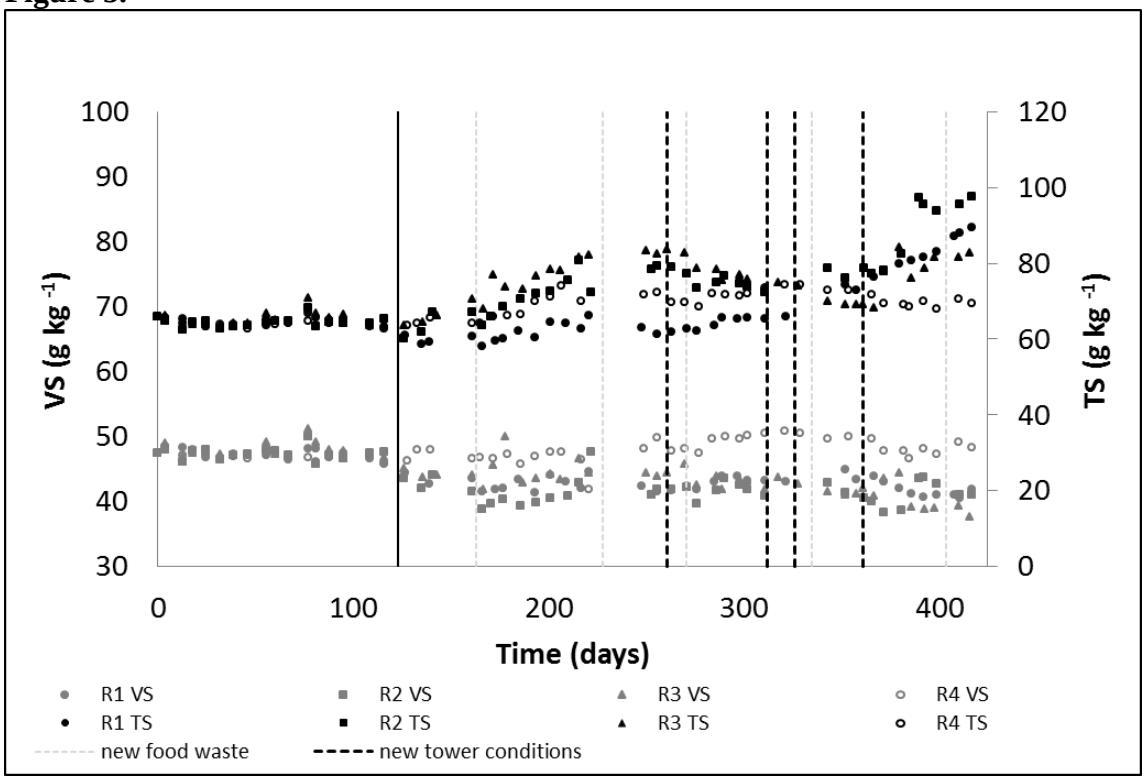

Figure 5. Digestate total and volatile solids concentrations during the experimental period (grey: principal axis - VS, black: secondary axis - TS). R_1: closed circle. R_2: closed square. R_3: closed triangle. R_4: open circle. Black continuous vertical line indicates start of stripping. Black discontinuous vertical lines indicate a change in stripping conditions (Table 2). Grey discontinuous vertical lines indicate a new batch of food waste (Table 1).