Effect of oil content on biogas production and performance stability of anaerobic digestion of food waste

Olumide Wesley Awe,[1,2,3*], Jiaxin Lu[2], Shubiao Wu[2], Yaqian Zhao[1], Ange Nzihou[3], Nathalie Lyczko[3], and Doan Pham Minh[3]

[1] UCD Dooge Centre for Water Resources Research, School of Civil Engineering, University College Dublin, Newstead, Belfield, Dublin 4, Ireland

[2] Key Laboratory for Clean Renewable Energy Utilization Technology of Ministry of Agriculture, College of Engineering, China Agricultural University, Beijing 100083, People's Republic of China

[3]Université de Toulouse, Mines Albi, CNRS UMR 5302, Centre RAPSODEE, Campus Jarlard, Albi, F-81013 cedex 09, France

*Corresponding author: Olumide Wesley Awe; E-mail: wesley.awe@ucdconnect.ie

Abstract: The primary cause of anaerobic digester failure or upset includes accumulation of inhibitory substances and intermediate products such as volatile fatty acids (VFAs), free ammonia (NH_3^+), and ammonium (NH_4^+). They are however required as essential nutrients for bacteria growth and used mainly by methanogenic for methane production and oxidation process. The full details of their effects on the process performance and stability are still not well understood despite many studies on this subject. The current study specifically investigated the effect of oil content addition on the biogas production and performance stability of anaerobic digestion of food waste. Two lab scale reactors were designed with different organic loading rates (OLRs) and feeding adjustment of used oil addition to test the effects of lipids on biodegradation and biogas production. The results indicate that, at 2.0 g VS (L/d), the addition of oil (5% v/v), caused the reactor failure, whereas at 4.0 g VS (L/d), the reactor remained stable for 10 days before the accumulation of VFAs, which resulted in low pH, and thus reduced the biogas and methane production. The addition of NaOH to rescue the reactors can only improve pH, alkalinity and negatively increased viscosity, but no significant effect on biogas production and VFAs concentration. An effective solution to reactivate the reactors was achieved by recirculating 50% of both reactor's effluent back to the reactors. This resulted in biogas recovery and stable process performance of the reactors. Surprisingly, NH_4^+-N remained stable (1400 mg/L) throughout the period, far less than the critical concentration of 3000 mg/L. On the contrary, the low NH_4^+-N could contribute to buffer the reactor's high VFA concentration during the unstable period, thereby raising new questions on it roles in anaerobic digestion (AD) process.

Keywords: Biogas, Anaerobic co-digestion, Inhibition, NaOH dosing, Food Waste, Lipids.

1. INTRODUCTION

It has been reported that more than 1.3 billion tons of FW are discarded every year, with about 100 million tonnes coming from Europe [1,2] and Chinese landfill about 60 million tons of FW, with Beijing alone generating 1,600 tons per day [3]. According to the Food and Agriculture Organisation of the United Nation (FAO) [1,4], the food waste and losses amounts to roughly US$ 680 billion in industrialized countries and over US$ 310 billion in developing countries. This also amount to wasting of resources used in producing the loss and wasted food, which includes land, water, labour and capital, energy, and the unnecessary production and emission of GHGs, thereby contributing to global warming and climate changes [1,5,6] According Natural Resources Defense Council (NRDC) issued paper [7], the USA alone, more than 36.4 million tons of FW was sent the landfills in 2012, costing $165 billion, and responsible for 16% of her methane emissions [5,8].

Food waste can be divided into three broad categories; lipids, proteins and carbohydrates, with the biodegradability or hydrolysis rate differs for each of them; Lipids < Proteins < carbohydrates [9,10]. However, proteins and carbohydrates decomposed faster and can be use than lipids. That is why lipids degradation is seen as a rate limiting step for food waste anaerobic digestion [9]. Lipids in FW is a mixture of vegetable oils and animal fats and the production of vegetable oils and animal fats account for about 160 million tons per year worldwide, and about 80% of this are used for human consumption [10]. In the same vein, Chinese FW is totally different from other countries [1,11], this may be due to dietary habit in China. Typically, Chinese FWs are rich in oil and salinity content. They are characterised by high amount of organic matter; lipids (22.8-31.5%), protein (14.7-28.6%), and carbohydrate dry matter [5]. Due to the high organic carbon and protein content in FW coupled with high lipid content of used oil waste, they can be collected and co-digested with either sewage sludge or animal manures, for energy and resource recovery.

The lipids content in FW will affect the digestion process due to excessive production of long chain fatty acids (LCFAs). This has been proved to be toxic to anaerobic bacteria community [2,12,13]. The levels at which LCFAs become toxic vary widely, depending on which acids forms, which are predominant in the digester. Also, the primary cause of NH_3 production and accumulation in the digester is the degradation of FW and oil, as they are rich in protein [14,6]. In the digester, NH_3 and ammonium ion (NH_4^+) are always present, as they are used as essential nutrients for the bacteria growth. Therefore, NH_4^+-N can inhibit the activity of methanogens and hence reduce the biogas production when they are present in high concentration [6,15]. In the literature, it seems that NH_3 was reported to be more inhibitory than NH_4^+-N due to its capacity to penetrate through the cell membranes [16,17]. There is an uncertainty to the acceptable limit at which NH_3 concentration becomes inhibitive in the digesters. Interestingly, Moestedt et al. [16], reported that, a mixture with higher percentage of food waste than sewage sludge may not likely have ammonia inhibition, due to the availability of higher carbon in the mix.

Theoretically, according to [18,19], lipid has the capacity to generate more methane than protein and carbohydrate. The methane yield of (1000 mL/g VS), proteins (480 mL/g VS) and, carbohydrate (373 mL/g VS) has been reported by researchers [13,20]. In his contribution, Alves [21], concluded that despite its limitations, lipids waste are ideal substrate for methane production with biogas production of 1.425 g/L (CH_4 of 69.5%), proteins 0.921 g/L (CH_4 of 68.8%), and carbohydrates 0.830 g/L (CH_4 of 50%). And with VS content 95-99%, if thickened to 5%, can be suitable to AD. But mono digestion of FOG is practically impossible because of high lipids concentration and production of LCFAs, which are known for inhibition of anaerobic microorganisms [1,22–27]. The LCFAs produced are toxic to hydrogen-producing bacteria and acetotropic and hydrogenotropic methanogenic and acetoclastic bacteria, even at low concentrations [1,13,20,25,28]. As a result, oil has been added by some researcher to enhance biogas production to their reactors, with report of problems. The LCFAs adsorption onto the biomass caused many operational challenges; digester foaming, flotation, biological bulking, odours, oxygen mass-transfer difficulties, increased effluent concentrations of organic matter, which may result in substrate and product transport limitation, clogging of gas collection and handling systems, blockage of pipes and pumps [9,1,13,23,29–33]. In the same vein, high concentration of lipid can cause process instability through sludge flocculation (biomass wash out), direct inhibition, VFAs overload, and physical fouling of equipment ([30,34].

Many other researchers have focused on finding solution to these operational challenges, such as anaerobic co-digestion of fat-rich matter with organic matter such as sewage sludge, farm manure, agricultural waste, organic fraction of municipal waste ([20,4,35–37]. Others have suggested the addition of adsorbent into the digesters such as fibres and bentonite powder ([34], and the use of novel anaerobic flotation reactors, separation of oil prior to AD operation [13,18,33], in which case, the HRT can be reduced. Finally, coupling of thermophilic and mesophilic reactors with H_2 production prior to recirculation for CH_4 production [38,39], while Nielsen [40] compared two-stage thermophilic (55 degree) anaerobic digestion with one-stage thermophilic (55 degree) digestion of cattle manure [40].

With the high amount of lipids in FW, it will therefore be necessary to seek some effective methods of eliminating the limitation for achieving high performance of FW AD. However, the crucial issues are to investigate and determine the limit, where oil addition or oil content in FW does not inhibit the AD process. This study aims to investigate the effect of organic loading rates (OLR), alkalinity, tVFA, pH and NH_4^+ on the biogas production and methane yield of anaerobic digestion of FW and, also to examine the effect of oil addition on the process performance and stability.

2. MATERIALS AND METHODS

2.1 Materials

The FW used was collected from the student's restaurants, China Agricultural University, Beijing. It was screened manually to remove non-biodegradable impurities such as, wastepaper, metal items, plastic, large bones, and non-biodegradable materials. The remaining waste was grounded with electrical blender with addition of an equal water volume for dilution to obtain 12.8-15 % wet weight of TS content. The homogenised FW was shared and stored in a refrigerator at 4 ^oC for immediate use, while the rest was stored -10 ^oC for long time use to prevent biological decomposition. When the frozen FWs are to be used, they are brought out and thaw in a refrigerator at 4 ^oC, 24 hours prior to use. The characteristics of the FW are summarised in Table 1.

The Inoculum used in this study was initially collected from a large-scale biogas plant located in Shun Yi district, Beijing, China. It was previously used in a lab-scale anaerobic digestion that was operated and used for pig manure digestion, for 300 days, under mesophilic condition (37 ± 1 ^oC). The inoculum in the digester was incubated (kept running) at 37 ± 1 ^oC to degas it and to ensure complete degradation of residual organic matter, while at the same time to remove the dissolved methane content. The characteristics of the inoculum are jointly shown in Table 1.

Table 1. Characterisation of substrates used for anaerobic digestion in two parallel CSTRs.

Parameters	Inoculu	Inoculum	Food Waste	FW+OIL

	m (R1)	(R2)	(FW)	(R1 & R2)
Ph	7.3	7.4	5.85	5.91
TS (%)	5.7	5.3	14.3	18.50
VS (%)	3.6	2.4	13.1	17.57
%VS (of TS)	64	53.5	91.90	94.99
Ash content (%)	33.71	46.61	8.01	8.01
NH_4^+-N (mg/L)	3805	651	166	-
TCOD (mg/L)	12,750	21,860	154,250	358,000
CODs (mg/L)	6,750	18,500	39,083	96,750
SCOD/TCOD	0.53	0.85	0.25	0.27
C (%)	21.50	29.74	51.12	57.02
N (%)	9.52	2.79	2.74	2.63
O (%)	29.08	16.08	30.41	23.84
H (%)	4.87	3.93	7.2	7.84
S (%)	1.33	0.77	0.52	0.66
C/N	2.26	10.66	18.68	21.68
Carbohydrate (g-glucose/L)	-	-	26.51	33.21
Lipids (g/L)	-	-	51.1	98.25

2.2 The CSTR experimental design and set up

Two laboratory-scale semi-continuous stirred-tank reactors (CSTRs) under mesophilic conditions, with 15 and 10 litres of total and effective working volumes, respectively, are employed in this study as shown in Fig. 1. There were two ports on each reactor; with feeding and effluent discharge port set at the top and bottom of each reactor, respectively. The biogas was collected with 60 L gas bag and connected at the top of each reactor. The two reactors were stirred intermittently by top-mounted mechanical stirrer at 120 rpm with 1 hour and 1 hour off to ensure total mix. The mesophilic ($37 \pm 1\ ^oC$) condition was maintained using temperature-controlled water baths (LML-1). Two OLRs were set for the two reactors. The first reactor called reactor (R1) OLR was set at 2.0 g VS (L/d) with 160 g of FW diluted to 500 ml, which amounted to 20 days' hydraulic retention time (HRT), while the second reactor called reactor (R2) OLR was set at 4.0 g VS(L/d) with 320 g of FW diluted to 500 ml, which amounted to 20 days' hydraulic retention time (HRT). These were fed into the reactors once per day and equal amount of effluent digestate were withdrawn manually per day and kept in a single container as mixed effluent. The experiment was divided into eight phases in order to investigate the effect of oil additions on various OLRs, biogas productions and methane yield, performance and stability of the entire process as well. Phase I (0-25), involved the addition of FW alone with the two mentioned OLRs, phase II (25-29) involved addition of 5 % oil content based on OLRs, phase III (29-30) oil addition was stopped, phase IV (30-40), reactor R1 OLR was changed to 4.0 g VS (L/d), phase V (40-57) 5% oil content was added again, phase VI (57-63) NaOH was added due to low PH and LCFA accumulation, phase VII (63-68), feed was stopped on both reactors for five days, and phase VIII (68-90), 50% recirculation of effluent was conducted, and biogas production restarted and continued to the end . The experiment ran for 90 days, and the residual digestate was analysed daily for pH, VS, TS, tVFA, alkalinity or total inorganic carbon (TIC), NH_4^+-N, soluble chemical oxygen demand (SCOD), total chemical oxygen demand (TCOD), as well as daily biogas production and methane yields.

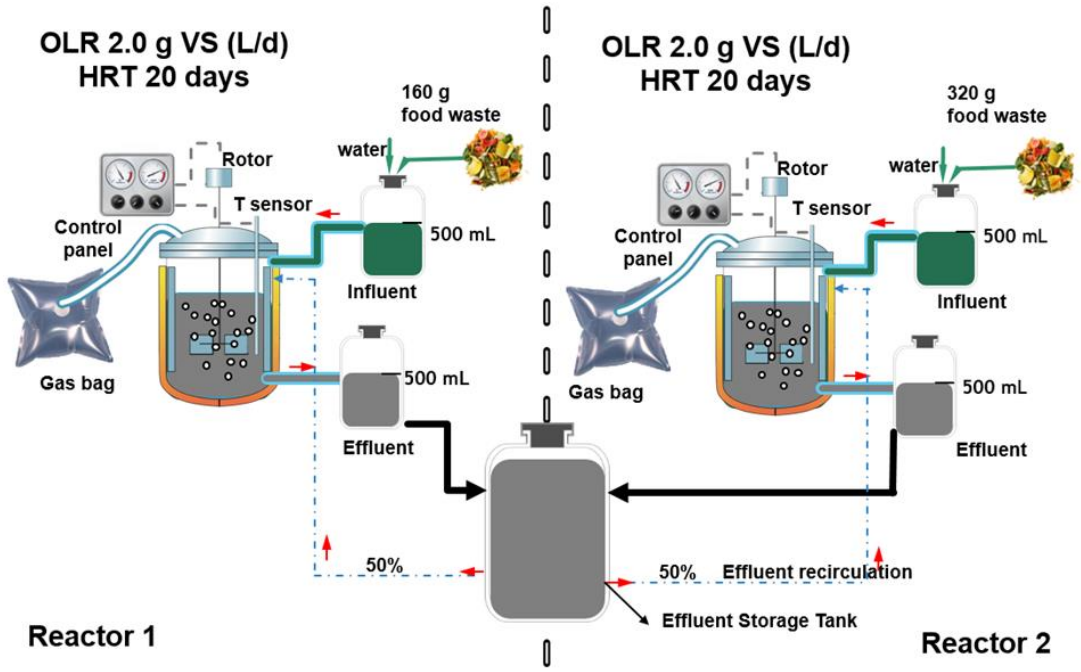

Fig. 1 Flow diagram of laboratory-scale setup for anaerobic digestion of food waste with effluent tank for recirculation

2.3 Analytical methods and calculations

The VS, TS, and NH_4^+-N, were determined following the standard methods of the American Public Health Association [41]. The PH was determined using a digital PH meter (FE20, METTLER TOLEDO, Switzerland) coupled with glass electrode (LE438, METTLER TOLEDO, Switzerland. To obtain the soluble fraction of the sample material, it was centrifuge (10,000 rpm for 15 munities), and the supernatant filtered through 0.45 µm cellulose acetate membrane. Part of the filtered samples was used to determine the NH_4^+-N concentration, using a spectrophotometer (UV-1100, MAPADA Instrument). The SCOD and TCOD were determined using a HATCH DR/2800 spectrometer (Hatch Company, USA) following the standard methods in APHA, 2005. The tVFA, total alkalinity or TIC content were analysed using Nordmann-titration method with 0.1N H_2SO_4 to endpoints of pH 5.0 and 4.4 [11,25,27]. The sample was centrifuge (10,000 rpm for 15 munities), diluted four times with deionized water and, 20 ml part of it was titrated, according to [9]. The tVFA/TIC (called FOS/TAC in Germany), as a digestion monitoring information were also determined. The values of TIC and TVFA were calculated using Nordmann [42], empirical equations (1) and (2):

$$\text{tVFA or FOS (mg/L)} = \left(\frac{20}{A}\right) * B * 1.66 - 0.15) * 500 \quad (1)$$

$$\text{TIC or TAC (mg/L } CaCO_3) = \left(\frac{20}{A}\right) * C * 250 \quad (2)$$

Where A is the volume of centrifuged sample used (ml), B is the volume of acid (0.1N H_2SO_4) used to go from pH 5 to pH 4.4 (ml), and C is the volume of acid (0.1N H_2SO_4) used to go from start to pH 5.5 (ml).

The two reactors inoculum samples, FW sample with and without oil addition were oven-dried (105 ^oC) and used for, ash content, ultimate analysis to determine carbon (C), nitrogen (N), sulphur (S) and hydrogen (H) content by elemental analyser (Vario EL/microcube, Germany). The value of Ash content and oxygen (O_2) were calculated using equations (3) and (4) as following:

$$\text{Ash (\%)} = \frac{Mash - Mcont}{Mod - Mcont} \times 100 \quad (3)$$

Where Ash (%), is the mass percent of ash, based on 105 ^oC oven-dried mass of sample, M_{ash} is the mass of ash and container (g), M_{cont} is the tare mass of container (g), and M_{od} is the initial mass of 105 ^oC dried sample and container (g).

$$\text{Oxygen } (O_2)\ \% = (100 - (C + N + S + H + Ash)) \quad (4)$$

The daily biogas production volume was measured with wet-type precision gas meter (LML-1, Changchun, China). The measured wet biogas and methane volumes were normalised and adjusted to the volumes at standard temperature

(213.15K) and pressure (101.325 kPa). The biogas composition was analysed by using BIOGAS 5000 portable biogas analyser (Geotechnical Instruments UK Ltd). The gas analyser was calibrated using certified gas, CH_4 (6%, 6%, 60), CO_2 (5%, 10%, 40%) and, O_2 (6%, 0%, 0%). The biogas and methane yields were calculated by dividing the daily gas yield (normalised), by the daily VS added to the reactors.

3 RESULTS AND DISCUSSION

3.1 Initial effects of oil addition to both reactors

This study investigated the effects of used oil content addition on the biogas production and performance stability of anaerobic co-digestion of food waste. Fig. 2 shown the biogas yield, methane yield (mL/g VS/d), and biogas composition. The two reactors were operated at 2.0 g VS (L/d) for R1, and 4.0 g VS (L/d) for R2 with stable operations for 25 days (phase I), after which oil content of (5% v/v), based on OLRs was added to the food waste and fed to both reactors. Expectedly, TCOD of the mixtures increased to 295,250 mg/L for the R1 and 304,000 mg/L for R2, respectively. After two days of operation (phase II), there was an acidification of the reactor (R1) as a result of the oil additions, due to high lipid concentrations and accumulation of LCFAs and because the R2 was still operating well with increased biogas production. This led to the conclusion, that reactor R1 was overloaded. This resulted in pH dropped from 6.9 to 6.1. This failure can probably also due to limited substrate and product transport or according to [11,43], damage cells and reduced activities of microbial communities which may have been stressed. Biomass floatation and biomass washout was observed with R1 effluent, with TCOD increased to 79,400 mg/L, while that of R2 remined at 37,600 mg/L.

3.2 Effects of first changes on the reactors

To rescue the reactor R1, firstly oil addition to both reactors were stopped. Secondly, by increasing the organic loading rate from 2.0 g VS (L/d) to 4.0 g VS (L/d) as reactor R2, since it was apparently clear that, at 2.0 g VS (L/d) OLR, the reactor was being overloaded with the 5% oil addition. Thirdly, we tried effluent recirculation from the single effluent storage tank, that received effluents from both reactors, with 40% withdrawal from reactors (R1) and 20% from (R2), and feeding the same quantities from the tank. The only notable changes in reactor R2 was the significant reduction in biogas production from 28 L/(L.d) to 9.2 L/(L.d) (26.39%), while that of reactor R1 was reduced from 9.9 L/(L.d) to 2.3 L/(L.d) (20.37%) and, reduced methane content of both. It worked, because the pH immediately increased and stabilised at 7.3 for both reactors (Fig. 5d) and biogas production significantly increased in reactor R1 from 2.3 L(L/d) to 23.7 L/(L.d) (90.30%) and in reactor R2 from 10 L/(L.d) to 25.2 L/(L.d) (63.49%). This were clearly shown in Fig. 2 (a, b and c).

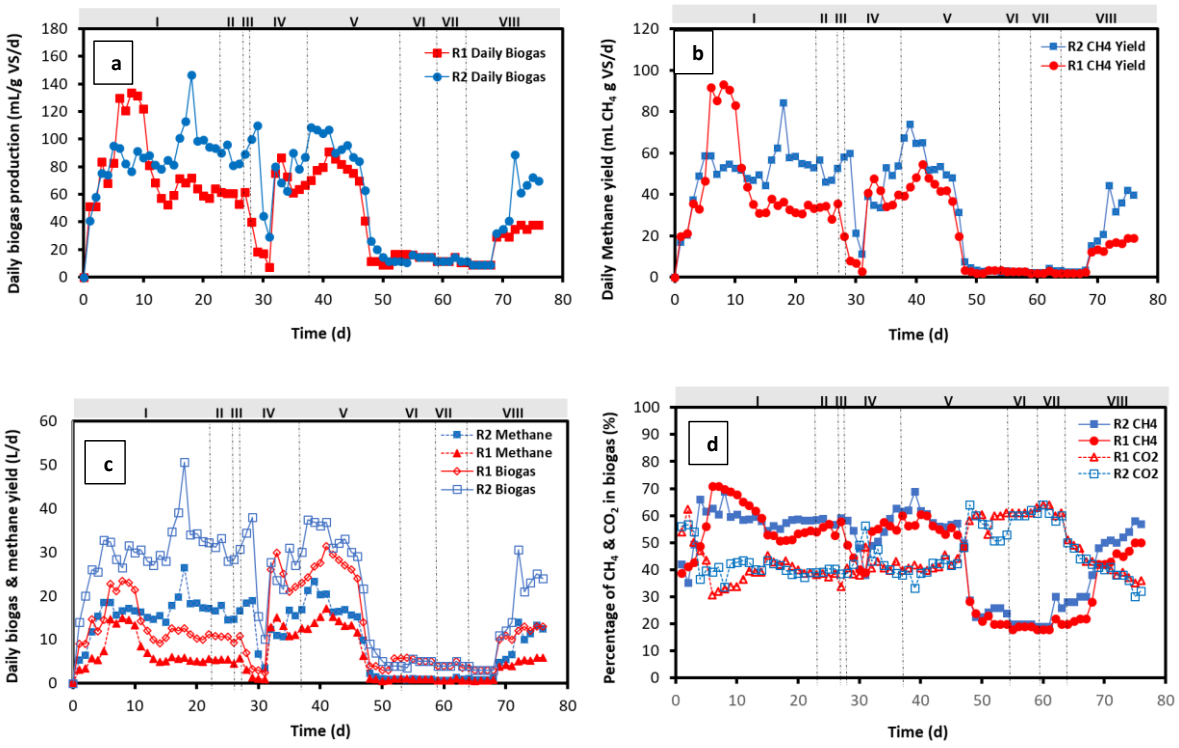

Fig. 2 The dynamic changes of (a) daily biogas production (mL/g VS/d), and (b) daily methane yield (mL CH_4 g VS/d), (c) daily biogas and methane yield (L/d), and (d) biogas composition in the process of FW anaerobic digestion under mesophilic condition

3.3. Effects of oil addition on the process performance and stability of the reactors

After further 40 days of stable operation with stable reactors, and after initial 15 days' downtime and recovery, FW was processed and analysed for all the parameters including lipid test to ascertain the amount of lipid present in the FW before the addition of 5% oil based on the reactor's OLRs. This runs successfully for the first 8 days (day 47th), with TCOD conversion efficiency peaking at 95% for R1 and 94% for R2 (Fig. 3d). The biogas production also increased, peaking at 28.6 L/(L.d) for R1, and 33.6 L/(L.d) for R2 (Fig. 2c). The peak CH_4 content for R1 was 60% as shown in Fig. 2d, which yield 17.15 L/d, while R2 CH_4 content was 61%, yielding 20.43 L/(L.d). Other parameters analysed (tVFAs, TIC, FOS/TAC, NH_4^+-N, VS and TS), shows a clear stable reactor operations.

3.4 Effects of oil addition on SCOD, TCOD, TCOD conversion efficiency, and effluent quality

Thereafter, the pH, as shown in the figure, started to reduce (R1 from 7.2 to 6.3 and R2 from 7.4 to 6.9). However, feeding continue without oil addition to see how far this effect will go, until the pH finally reached 5.1 for R1 and 5.2 for R2, with TCOD conversion efficiency (Fig. 3d) reduced to 17% for R1 and 41% for R2, and biogas production reduced significantly to 3.7 L/(L.d) for both reactors. This represents a reduction of 87.06% and 88.99% for R1 and R2, respectively. There are noticeable effluent colour changing from deep black to brownish colour with lipids formations and foaming. This might be due to the inability of the microorganisms not being able to utilise the fed substrate, as a result of the inhibitions posed by the LCFAs accumulation in the reactor which has become toxic for the microorganisms. All these negative effects have led to increase in odour level of the effluent.

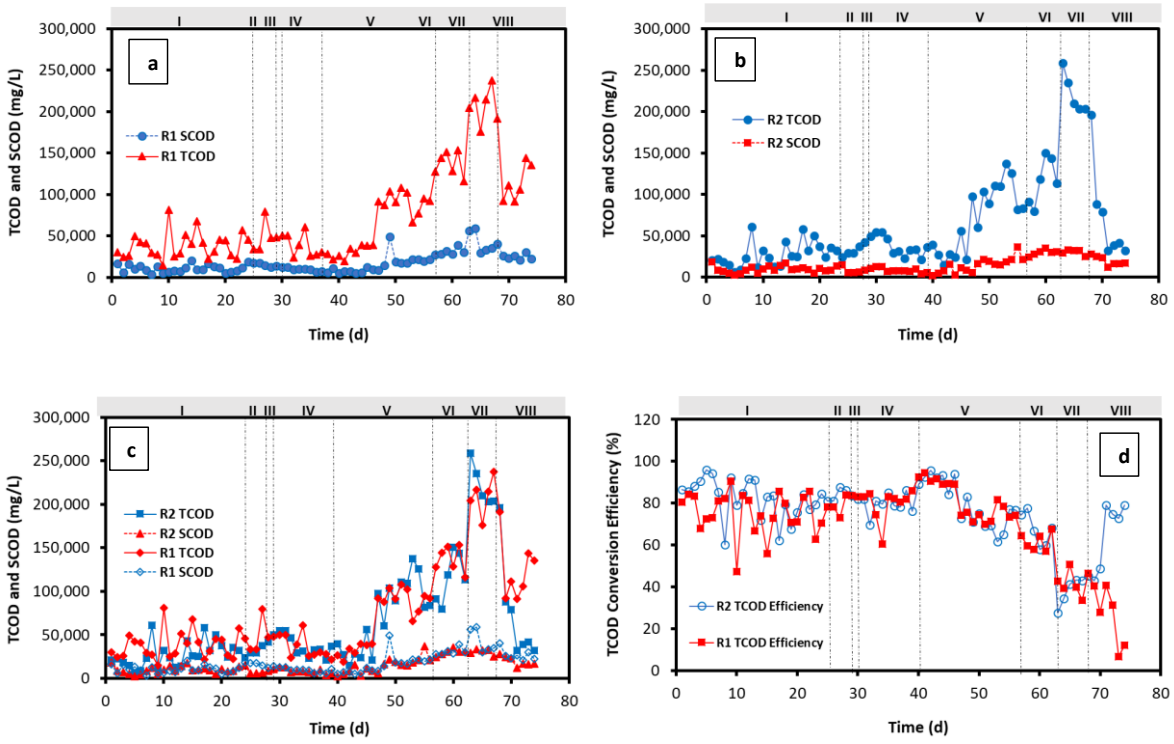

Fig. 3 The dynamic changes of (a) R1 SCOD and TCOD, (b) R2 SCOD and TCOD, (c) combining the two reactors, (d) and TCOD removal efficiency for reactors R1 and R2, in the process of FW anaerobic digestion under mesophilic condition.

3.5 Effects of oil addition on pH, tVFAs, TIC and FOS/TAC

Fig. 4 shows the dynamisms AD of food waste without and with oil addition. It can be seen that, tVFAs increased significantly for R1 from the initial 1526 mg/L when oil was added and FOS/TAC ratio of 0.55, to 4846 mg/L and FOS/TAC ratio of 10.77, while R2 increased astronomically from initial 364 mg/L when oil was added and FOS/TAC ratio of 0.10, to 5012 mg/L and FOS/TAC ratio of 10.77. The changes are clearly shown in Fig 4d. This represents an increase of 68.51% and 92.73% for reactors R1 and R2 respectively, and lead to both reactor failures. It has been shown by Raposo [44] that, a drop in PH, leads to increase in VFA/TIC ratio (>0.4), as result of high VFA concentrations. Thus, the failure of the reactors is in agreement with [44,45] finding that, operation of the AD will cease

to be stable, at a higher FOS/TAC ratio above >0.5. On the other hands, the buffering capacity (alkalinity) of the reactors reduced significantly for R1, from the initial 2750 mg/L $CaCO_3$ when oil was added to 200 mg/L $CaCO_3$, a reduction of 92.73%, while that of R2, reduced from the initial 3500 mg/L $CaCO_3$ when oil was added to 300 mg/L $CaCO_3$, a reduction of 91.43% as shown in Fig.4a. On the other hands, Fig. 4c shows the combined effects of alkalinity (TIC) and tVFA on the two reactors. From Fig. 4b and 4c, there is a corresponding effect of high VFA concentration on the reduction of the pH values, which definitely have negative effects on the activities of methanogenesis. This persisted in phase (V) up to phase (VII), despite the addition of NaOH to increase the buffering capacity (alkalinity) and the pH.

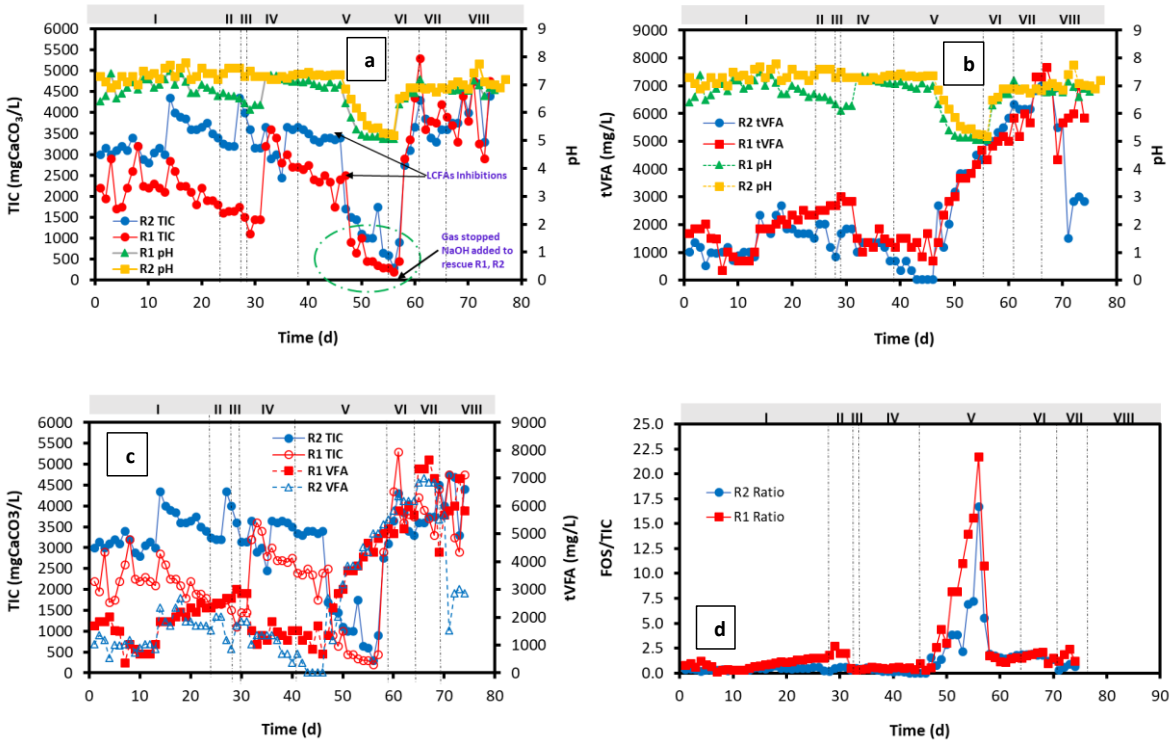

Fig. 4 The dynamic changes of (a) alkalinity (TIC) with PH, (b) tVFA with PH, (c) combining alkalinity (TIC) and tVFA for reactor R1and reactor R2, (d) FOS/TAC ratio in the process of FW anaerobic digestion under mesophilic condition

3.6 Effects of oil addition on NH_4^+-N, TS reduction, VS conversion efficiency, and pH

Fig. 5c shows the effects NH_4^+-N and surprisingly, NH_4^+-N has been relatively stable even after the addition of 5% oil contents to the reactors, with NH_4^+-N ranging between 805 mg/L to 1404 mg/L for R1 and, 797 mg/L to 1558 mg/L for R2. This is despite the reported protein content (14.7-28.6%) of Chinese FW [5], and source of nitrogen in FW. Despite no ammonia nitrogen inhibition, the system still failed and no biogas produced. This called for new question on the role of free ammonia or ammonium on process performance and stability of AD, when there are high VFA concentration in the digester. It has already been established that, ammonia plays a significant role in carbon to nitrogen (C/N) ratio balancing. In this study, the analysed C/N for FW alone was 18.68 and 21.68 for FW with 5% oil additions, which in agreement with works of [5,1,38]. Some researchers have suggested that, ammonia can also buffer or neutralised the VFAs form during the anaerobic digestion process, thereby stabilising the system and avoid AD failure [1,6,43,46]. As shown in Fig. 5c, the tolerable or critical ammonium concentration for methanogenic microorganisms to stop growing or react negatively is 3000 mg/L. And in this study, NH4+-N concentration fell in the range of 770-2748 mg/L for R1, and 325-1600 mg/L for R2, which is lower than the critical NH4+-N concentration of 3000 mg/L. This is similar with the work of [1], with range of 476.9-1645 mg/L. And despite this, the biogas production continued reducing significantly toward the end of phase (V), and up to early part of phase (VIII). This study result, is not also in agreement with the findings of [47], who reported a higher ammonia concentration of more than 5000 mg/L, during similar semi-continuous of anaerobic digestion of food waste. The R1 TS reduction efficiency stood at 83% after the oil was added, but later reduced to 63%, while that of R2 reduced from 85% to 61% within the same period. These changes are shown in Fig. 5 (a). This downward trend continued to 59% for both reactors until recovery. The effects clearly manifest in the physical appearance of the effluent with more TS concentration due to low conversion of the substrate by the reactors. It is possible that the high VFA concentration might have prevented or reduce the mass transfer ability, which some researchers [1,32,48,49] have identified, hence the high viscosity of the effluent. The same trend was observed with VS destruction efficiency (Fig. 5b), which reduced for R1, from 87% when oil was added to 60%, while R2 destruction

efficiency reduced from 87% to 67%. Strangely, this 60% and 67% destruction efficiency does not translate into biogas production. These is need to investigate why a 60% and 67% destruction efficiency does not necessarily translate into biogas production, because both reactors remained stagnated at 4 L/(L.d), until recovery process took effects.

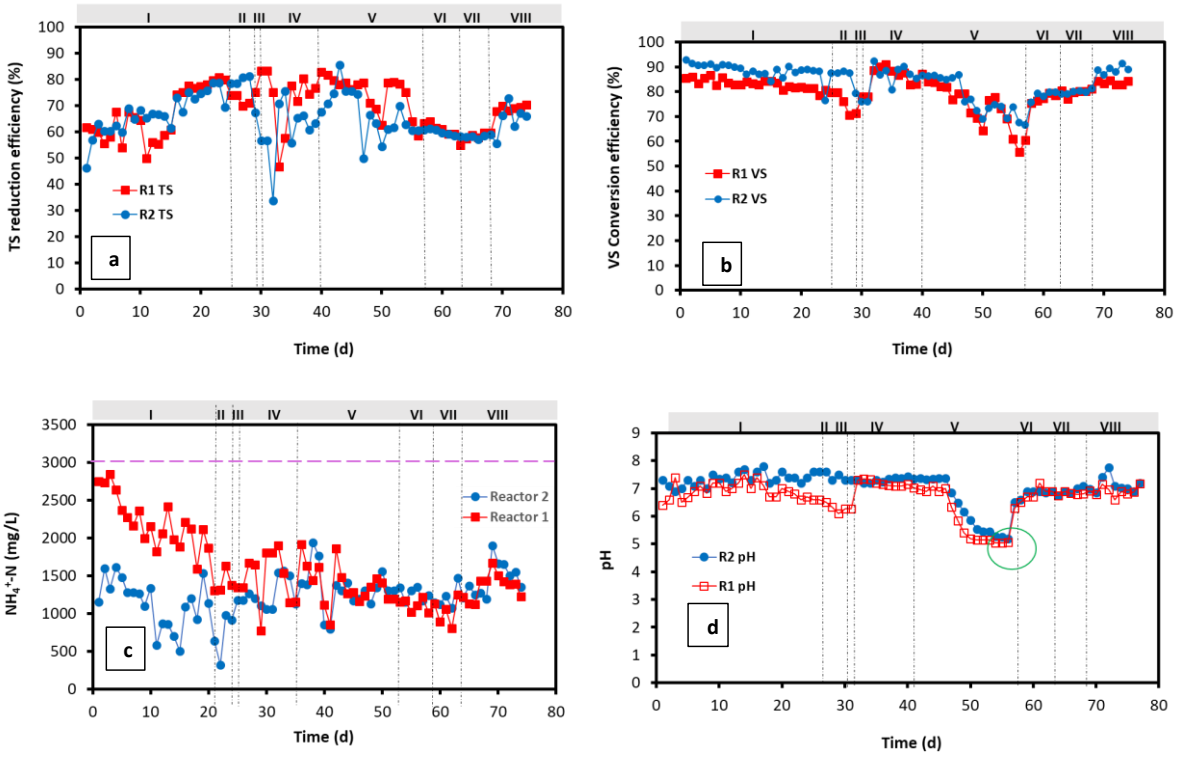

Fig. 5

The dynamic changes of (a) TS destruction efficiency, (b) VS conversion efficiency, (c) ammonium (NH_4^+-N), and (d) PH, in the process of FW anaerobic digestion under mesophilic condition

3.7 Effects of NaOH addition on to the reactors recovery

When the system failed the second time, the authors decided to rescue the reactors in stages. Firstly, a standard solution of 3 mol/L NaOH was used to adjust the pH [11]. Titration method was employed in order to know what quantity of NaOH to add to each reactor in order to increase the pH. About 250 ml (2.5% v/v) were added to the two reactors for the first three days during feeding, and later reduced to 50 ml (0.5% v/v) for three additional days afterward and stopped when the pH stable at 7.1 ± 0.1 for R1 and 7.2 ± 0.1 for R2 respectively (Fig. 4d). Apart from pH recovery, alkalinity (buffering capacity) has also increased due to the addition of NaOH for R1, from 200 mg/L $CaCO_3$ to 5,300 mg/L $CaCO_3$ (96.23%) and R2 from 300 mg/L $CaCO_3$ to 4,300 mg/L $CaCO_3$ (93.02%) as shown. Even though TIC has already increased but, it has no effect to buffer the high VFAs concentration probably due to low pH levels of both reactors. The question now is, why is VFAs not reducing with the increased alkalinity, which its major role is to buffer the acids and keep the reactor safe? This needs further investigation.

3.8 Effect of stopped feeding on the reactors recovery

The VFAs was on steady increase, thereby reducing the amount of biogas produced to less than 3.7 L/(L.d). This may have been caused by the stressed microorganism not been able to consume the substrate supplied due to high concentration of VFAs. Therefore, the second stage of the rescue was to stop feeding completely for at least 5 days, so that the methanogenic microorganisms can recover to consume and convert the accumulated VFAs in the reactors to biogas production. The two reactors effluent is smooth-milky substance with little foams, with increased in viscosity especially of reactor R2. This may have been caused by the dissolution of the accumulated LCFAs as a result of the addition of NaOH to the reactors, coupled with daily feeding that is not been utilised by the microorganisms. This had great effect on the effluent viscosity and increased TS concentrations.

3.9 Effect of effluent recirculation effects on the reactors recovery

After observing the effects of NaOH addition without any recovery in biogas production, coupled with high VFA concentration continue increase, 50% of the stored effluent were used by withdrawing the same amount from the reactor prior to the recirculation. This had an immediate effect on the biogas production and reduction in the VFA concentrations in both reactors. The characteristics of the effluent used in the recirculation and recovery are shown in Table 2, while the

appreciable biogas recovery can be shown in Fig. 2. The effect on the VFA can be seen in Fig. 4a and b. There was also increase in TS reduction, VS and TCOD conversion efficiency as shown in the figures. The withdrawn effluent can be gradually mixed with the FW and added to the reactors without any inhibitions.

Table 2. Analysis of the buffer tank effluent used to reactivate the two reactors

	TS (%)	**VS (%)**	**VS/TS (%)**	**SCOD (mg/L)**	**TCOD (mg/L)**	**NH_4^+-N (mg/L)**	**TIC mg/L$CaCO_3$**	**TVFA (mg/L)**	**FOS/TOAC**
Inoculum	4.64	3.04	65.56	7,000	38,750	2107	4,300	364	0.08

4 Conclusion

The results showed that lipids inhibitions and other operational challenges of oil addition to FW could be addressed with the combination of NaOH addition coupled with recirculation of certain percentage of the digester's effluent, to kick-start dilution and breaking the LCFAs, gradual reduction in the concentration of VFA from 6838 mg/L before recirculation to 2854 mg/L after 7 days of recirculation, and for quick resumption of biogas production. The biogas and production increased from 3 L/(L.d) before recirculation to 13 L/(L.d) for reactor R1 and from 3 L/(L.d) to 25 L/(L.d), while the methane production increased for reactor R1, from 0.77 L/(L.d) before recirculation to 5.94 L/(L.d), and from1.04 L/(L.d) to 13.24 L/(L.d) for reactor R2. This study provided understanding of the dynamic complex nature of Chinese food waste, especially because of it high salinity and lipids contents. In this study, ammonia was not a key inhibiting factor of FW in anaerobic digestion, with stable concentration of 1400 mg/L throughout the period, far less than the critical concentration of 3000 mg/L.

Acknowledgements

The first author acknowledges the support from School of Civil Engineering, University College Dublin; Tuition scholarship support from Student Universal Support Ireland (SUSI); Centre RAPSODEE, Campus Jarlard, Albi, F-81013 cedex 09, France and China Agricultural University, Key Laboratory of Clean Utilization Technology for Renewable Energy, Ministry of Agriculture. The author gratefully acknowledges colleagues and supervisor's contributions.

References

[1] W. Zhang, Q. Lang, M. Fang, X. Li, H. Bah, H. Dong, R. Dong, Bioresource Technology Combined effect of crude fat content and initial substrate concentration on batch anaerobic digestion characteristics of food waste, Bioresour. Technol. 232 (2017) 304–312. doi:10.1016/j.biortech.2017.02.039.

[2] R. Chen, Xiguang; Romano, Rowena T; Zhang, Anaerobic digestion of food wastes for biogas production, Int. J. Agric. Biol. Eng. 3 (2010) 61–72. https://ijabe.org/index.php/ijabe/article/view/214.

[3] Y. Sun, D. Wang, J. Yan, W. Qiao, W. Wang, T. Zhu, Effects of lipid concentration on anaerobic co-digestion of municipal biomass wastes, Waste Manag. 34 (2014) 1025–1034. doi:10.1016/j.wasman.2013.07.018.

[4] P.F. Gamble, R. Zhang, H.M. El-mashad, K. Hartman, F. Wang, Research Into Design and Modeling of Anaerobic Digestion Process Applied to Municipal Solid Wastes, (2015). doi:10.1016/j.biortech.2006.02.039.

[5] Y. Meng, S. Li, H. Yuan, D. Zou, Y. Liu, B. Zhu, A. Chufo, M. Jaffar, X. Li, Evaluating biomethane production from anaerobic mono- and co-digestion of food waste and floatable oil (FO) skimmed from food waste, Bioresour. Technol. 185 (2015) 7–13. doi:10.1016/j.biortech.2015.02.036.

[6] H. Wang, I.A. Fotidis, I. Angelidaki, Ammonia effect on hydrogenotrophic methanogens and syntrophic acetate-oxidizing bacteria, FEMS Microbiol. Ecol. 91 (2015). doi:10.1093/femsec/fiv130.

[7] D. Gunders, Wasted: How America is losing up to 40 percent of its food from farm to fork to landfill, NRDC Issue Pap. (2012) 1–26. doi:12-06-B.

[8] Epa, Inventory of U.S. Greenhouse Gas Emissions and Sinks: 1990-2010, Www.Epa.Gov. (2012) 1–481. doi:EPA 430-R-12-001.

[9] H. Sun, S. Wu, R. Dong, Monitoring Volatile Fatty Acids and Carbonate Alkalinity in Anaerobic Digestion: Titration Methodologies, Chem. Eng. Technol. 39 (2016) 599–610. doi:10.1002/ceat.201500293.

[10] C.S.K. Lin, L.A. Pfaltzgraff, L. Herrero-Davila, E.B. Mubofu, S. Abderrahim, J.H. Clark, A.A. Koutinas, N. Kopsahelis, K. Stamatelatou, F. Dickson, S. Thankappan, Z. Mohamed, R. Brocklesby, R. Luque, Food waste as a valuable resource for the production of chemicals, materials and fuels. Current situation and global perspective, Energy Environ. Sci. 6 (2013) 426. doi:10.1039/c2ee23440h.

[11] T. Zhang, C. Mao, N. Zhai, X. Wang, G. Yang, Influence of initial pH on thermophilic anaerobic co-digestion of swine manure and maize stalk, Waste Manag. 35 (2015) 119–126. doi:10.1016/j.wasman.2014.09.004.

[12] Y. Chen, J.J. Cheng, K.S. Creamer, Inhibition of anaerobic digestion process: A review, Bioresour. Technol. 99 (2008) 4044–4064. doi:10.1016/j.biortech.2007.01.057.

[13] H.S. Cho, H.S. Moon, J.Y. Lim, J.Y. Kim, Effect of long chain fatty acids removal as a pretreatment on the anaerobic digestion of food waste, J. Mater. Cycles Waste Manag. 15 (2013) 82–89. doi:10.1007/s10163-012-0092-7.
[14] B. Wang, I.A. Nges, M. Nistor, J. Liu, Determination of methane yield of cellulose using different experimental setups, Water Sci. Technol. 70 (2014) 599–604. doi:10.2166/wst.2014.275.
[15] I.A. Fotidis, D. Karakashev, T.A. Kotsopoulos, G.G. Martzopoulos, I. Angelidaki, Effect of ammonium and acetate on methanogenic pathway and methanogenic community composition, FEMS Microbiol. Ecol. 83 (2013). doi:10.1111/j.1574-6941.2012.01456.x.
[16] J. Moestedt, B. Müller, M. Westerholm, A. Schnürer, Ammonia threshold for inhibition of anaerobic digestion of thin stillage and the importance of organic loading rate, Microb. Biotechnol. 9 (2016). doi:10.1111/1751-7915.12330.
[17] R. Rajagopal, D.I. Massé, G. Singh, A critical review on inhibition of anaerobic digestion process by excess ammonia, Bioresour. Technol. 143 (2013). doi:10.1016/j.biortech.2013.06.030.
[18] M.. Alves, J.. Mota Vieira, R.. Álvares Pereira, M.. Pereira, M. Mota, Effects of lipids and oleic acid on biomass development in anaerobic fixed-bed reactors. Part II: Oleic acid toxicity and biodegradability, Water Res. 35 (2001) 264–270. doi:10.1016/S0043-1354(00)00242-6.
[19] S. Astals, D.J. Batstone, J. Mata-Alvarez, P.D. Jensen, Identification of synergistic impacts during anaerobic co-digestion of organic wastes, Bioresour. Technol. 169 (2014) 421–427. doi:10.1016/j.biortech.2014.07.024.
[20] L. Pastor, L. Ruiz, A. Pascual, B. Ruiz, Co-digestion of used oils and urban landfill leachates with sewage sludge and the effect on the biogas production, Appl. Energy. 107 (2013) 438–445. doi:10.1016/j.apenergy.2013.02.055.
[21] M.M. Alves, M.A. Pereira, D.Z. Sousa, A.J. Cavaleiro, M. Picavet, H. Smidt, A.J.M. Stams, Waste lipids to energy: How to optimize methane production from long-chain fatty acids (LCFA), Microb. Biotechnol. 2 (2009) 538–550. doi:10.1111/j.1751-7915.2009.00100.x.
[22] N. Rasit, A. Idris, R. Harun, W.A. Wan Ab Karim Ghani, Effects of lipid inhibition on biogas production of anaerobic digestion from oily effluents and sludges: An overview, Renew. Sustain. Energy Rev. 45 (2015). doi:10.1016/j.rser.2015.01.066.
[23] Å. Davidsson, C. Lövstedt, J. la Cour Jansen, C. Gruvberger, H. Aspegren, Co-digestion of grease trap sludge and sewage sludge, Waste Manag. 28 (2008) 986–992. doi:10.1016/j.wasman.2007.03.024.
[24] J. Kim, C.-M. Kang, Increased anaerobic production of methane by co-digestion of sludge with microalgal biomass and food waste leachate, Bioresour. Technol. 189 (2015) 409–412. doi:http://dx.doi.org/10.1016/j.biortech.2015.04.028.
[25] G.K. Kafle, S.H. Kim, B.S. Shin, Anaerobic Digestion Treatment for the Mixture of Chinese Cabbage Waste Juice and Swine Manure, 37 (2012) 58–64.
[26] G. Luo, I. Angelidaki, Co-digestion of manure and whey for in situ biogas upgrading by the addition of H2: process performance and microbial insights, Appl. Microbiol. Biotechnol. 97 (2012) 1373–1381. doi:10.1007/s00253-012-4547-5.
[27] D.G. Cirne, X. Paloumet, L. Björnsson, M.M. Alves, B. Mattiasson, Anaerobic digestion of lipid-rich waste—Effects of lipid concentration, Renew. Energy. 32 (2007) 965–975. doi:10.1016/j.renene.2006.04.003.
[28] M. Kim, Y.H. Ahn, R.E. Speece, Comparative process stability and efficiency of anaerobic digestion; mesophilic vs. thermophilic, Water Res. 36 (2002). doi:10.1016/S0043-1354(02)00147-1.
[29] J. Mata-Alvarez, J. Dosta, M.S. Romero-Güiza, X. Fonoll, M. Peces, S. Astals, A critical review on anaerobic co-digestion achievements between 2010 and 2013, Renew. Sustain. Energy Rev. 36 (2014) 412–427. doi:10.1016/j.rser.2014.04.039.
[30] J.H. Long, T.N. Aziz, F.L.D.L. Reyes, J.J. Ducoste, Anaerobic co-digestion of fat, oil, and grease (FOG): A review of gas production and process limitations, Process Saf. Environ. Prot. 90 (2012) 231–245. doi:10.1016/j.psep.2011.10.001.
[31] S. Alanya, Y.D. Yilmazel, C. Park, J.L. Willis, J. Keaney, P.M. Kohl, J.A. Hunt, M. Duran, Anaerobic co-digestion of sewage sludge and primary clarifier skimmings for increased biogas production., 67 (2013) 174–9. doi:10.2166/wst.2012.550.
[32] L.D. Nghiem, K. Koch, D. Bolzonella, J.E. Drewes, Full scale co-digestion of wastewater sludge and food waste : Bottlenecks and possibilities, Renew. Sustain. Energy Rev. 72 (2017) 354–362. doi:10.1016/j.rser.2017.01.062.
[33] N. Rasit, A. Idris, R. Harun, W.A. Wan Ab Karim Ghani, Effects of lipid inhibition on biogas production of anaerobic digestion from oily effluents and sludges: An overview, Renew. Sustain. Energy Rev. 45 (2015) 351–358. doi:10.1016/j.rser.2015.01.066.
[34] J. Palatsi, M. Viñas, M. Guivernau, B. Fernandez, X. Flotats, Anaerobic digestion of slaughterhouse waste: Main process limitations and microbial community interactions, Bioresour. Technol. 102 (2011) 2219–2227. doi:10.1016/j.biortech.2010.09.121.
[35] H. V Hendriksen, B.K. Ahring, Effects of ammonia on growth and morphology of thermophilic hydrogen-oxidizing methanogenic bacteria, FEMS Microb Ecol. 85 (1991). doi:10.1111/j.1574-6968.1991.tb04730.x.

[36] H.B. Nielsen, H. Uellendahl, B.K. Ahring, Regulation and optimization of the biogas process: propionate as a key parameter, Biomass Bioenerg. 31 (2007). doi:10.1016/j.biombioe.2007.04.004.
[37] L. Alibardi, R. Cossu, Composition variability of the organic fraction of municipal solid waste and effects on hydrogen and methane production potentials, Waste Manag. 36 (2015) 147–155. doi:10.1016/j.wasman.2014.11.019.
[38] D.E. Algapani, W. Qiao, M. Su, S.M. Wandera, F. Adani, Bioresource Technology Bio-hydrolysis and bio-hydrogen production from food waste by thermophilic and hyperthermophilic anaerobic process, Bioresour. Technol. 216 (2016) 768–777. doi:10.1016/j.biortech.2016.06.016.
[39] C. Cavinato, D. Bolzonella, P. Pavan, F. Fatone, F. Cecchi, Mesophilic and thermophilic anaerobic co-digestion of waste activated sludge and source sorted biowaste in pilot- and full-scale reactors, Renew. Energy. 55 (2013) 260–265. doi:10.1016/j.renene.2012.12.044.
[40] H.B. Nielsen, Z. Mladenovska, P. Westermann, B.K. Ahring, Comparison of two-stage thermophilic (55 degree) anaerobic digestion with one-stage thermophilic (55 degree) digestion of cattle manure, Biotechnol Bioeng. 86 (2004). doi:10.1002/bit.20037.
[41] APHA, Standard methods for the examination of water and wastewater., Public Health Association Ic, Washington DC, 2005.
[42] W. Nordmann, Die Überwachung der Schlammfaulung, KA-Informationen Für Das Betriebspersonal, Beilage Zur Korrespondenz Abwasser. 3 (1977) 77.
[43] B. Wang, S. Strömberg, C. Li, I.A. Nges, M. Nistor, L. Deng, J. Liu, Effects of substrate concentration on methane potential and degradation kinetics in batch anaerobic digestion, Bioresour. Technol. 194 (2015) 240–246. doi:10.1016/j.biortech.2015.07.034.
[44] F. Raposo, M.A.D. Rubia, V. Fernandez-Cegri, R. Borja, Anaerobic digestion of solid organic substrates in batch mode: an overview relating to methane yields and experimental procedures, Renew Sustain Energy Rev. 16 (2011). doi:10.1016/j.rser.2011.09.008.
[45] R. Borja, B. Rincón, F. Raposo, J.R. Domínguez, F. Millán, A. Martín, Mesophilic anaerobic digestion in a fluidised-bed reactor of wastewater from the production of protein isolates from chickpea flour, Process Biochem. 39 (2004) 1913–1921. doi:10.1016/j.procbio.2003.09.022.
[46] C.J. Banks, M. Chesshire, S. Heaven, R. Arnold, Anaerobic digestion of source-segregated domestic food waste: Performance assessment by mass and energy balance, Bioresour. Technol. 102 (2011) 612–620. doi:10.1016/j.biortech.2010.08.005.
[47] C.J. Banks, Y. Zhang, Y. Jiang, S. Heaven, Trace element requirements for stable food waste digestion at elevated ammonia concentrations, Bioresour. Technol. 104 (2012) 127–135. doi:10.1016/j.biortech.2011.10.068.
[48] H. Sun, S. Wu, R. Dong, Monitoring Volatile Fatty Acids and Carbonate Alkalinity in Anaerobic Digestion: Titration Methodologies, Chem. Eng. Technol. 39 (2016) 599–610. doi:10.1002/ceat.201500293.
[49] N. Rasit, A. Idris, R. Harun, W.A. Wan Ab Karim Ghani, Effects of lipid inhibition on biogas production of anaerobic digestion from oily effluents and sludges: An overview, Renew. Sustain. Energy Rev. 45 (2015) 351–358. doi:10.1016/j.rser.2015.01.066.