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Two-Phase Anaerobic Digestion of Mixed Waste Streams to Separate Generation of Bio-hydrogen and Bio-methane

Z. Siddiqui, N.J. Horam, School of Civil Engineering, University of Leeds, W. Yorkshire, UK

Abstract

The purpose of this study was to investigate the net energy potential of single stage mesophilic reactor and two phase mesophilic reactor (hydrogeniser followed by methaniser) using the mix of process industrial food waste (IFW) and sewage sludge (SS). Two-phase reactor efficiency was analysed based on individual optimum influent/environmental (C:N and pH) and reactor/engineering (HRT and OLR) conditions achieved using the batch and continuous reactor study for the hydrogen and methane. Optimum C:N 20 and pH 5.5 ± 0.5 was observed using the Bio-H₂ potential (BHP) and C:N 15 and pH 6.5 ± 0.3 for the biochemical methane potential (BMP) test. The maximum hydrogen content of 47% (v/v) was achieved using OLR 6 g VS/L/d and HRT of 5 days. Increase in hydrogen yield was noticed with consistent decrease in OLR. The volatile solids (VS) removal and hydrogen yield was observed in range 41.3 to 47% and 112.3 to 146.7 mL/gVS_{removed}. The specific hydrogen production rate improved at low OLR, 0.2 to 0.4 L/(L.d) using OLR 7.1 and 6 g VS/L/d respectively was well corroborated comparable to previous reported results at OLR 6 gVS/L/d using the enriched carbohydrate waste stream in particular to food wastes. A significant increase in VFA concentrations were noticed shifting OLR higher from 6 g VS/L/d thereby unbalancing the reactor pH and the biogas yield respectively. In similar, maximum methane content of 70% (v/v) was achieved using OLR of 3.3 gVS/L/d and HRT of 10 days. Slight decrease in methane content was noticed thereby increasing HRT to 12 and 15 days respectively. The volatile solids (VS) removal and specific methane production rate was observed in range 57.6 to 68.7 and 0.22 to 1.19 L/(L.d). The specific methane production potential improved thereby reducing the HRT and optimum yield was recorded as 476.6 mL/gVS_{removed} using OLR 3.3 gVS/L/d. The energy potential of optimum condition in single stage hydrogeniser is 2.27 MW/tonne VS_{fed}. Using the two phase and sub optimal conditions improves the energy potential to 8.27 MW/tonne VS_{fed} with VS(removal) efficiency as 80.7% in total 15 days of HRT. The net energy balance results indicated the co-digestion of IFW with waste products of SS treatment plant viz. primary sludge (PS) and waste activated sludge (WAS) are amenable substrates for the two-stage anaerobic bio-hydrogen and bio-methane digestion process.

1 Introduction

A conventional phase-separated two-stage approach was implemented for biomethane production by introducing the hydrolyser/acidogeniser reactor in front of the methaniser (Pohland and Ghosh, 1971 [3]). This improves the buffering capacity of methaniser to feed shock-loads and to inhibitors such as ammonium and organic acids. It also improves the digester performance as the first phase accepts a higher loading in terms of OLR. The

production of biohydrogen is an attractive one as hydrogen is clean energy and a renewable energy production option for the future. However methane production has historically become more popular due to a lack of information on the process and complications involved in its optimisation. The results achieved from the batch and continuous reactor studies of biohydrogen and biomethane (Siddiqui, 2010 [2]) production that an integrated phase-separated two-stage anaerobic hydrogeniser and methaniser might be an ideal option for an optimised blend of organic feed-stocks. Whereas it was observed that a C:N 15 demonstrated optimum methane production (Siddiqui et al. 2010 [2]), the effluent characteristics of the hydrogeniser produced a C:N of 18 which may the methaniser for optimum methane production. One approach would be to have an intermediate feed directly to the methaniser to correct the imbalance, but initially it was decided to operate the methaniser under sub-optimal conditions by feeding the effluent from the hydrogeniser without correcting the C:N ratio. The aim of this study to investigate the net energy potential of single stage mesophilic reactor and two-phase mesophilic reactor (hydrogeniser followed by methaniser) using the mix of process industrial food waste (IFW) and sewage sludge (SS).

2 Material and Methods

2.1 Feedstock and seed inoculum

Feedstocks and nutrient supplements were selected from the earlier study described in Siddiqui (2010 [2]) with an ideal blend of carb:pro 2.78 (C:N 20) and pH 5.5 ± 0.3 selected and employed. The required feed with a blend of C:N 20 was achieved by mixing fractions of processed industrial food waste IFW, sewage sludge SS (primary sludge PS and waste activated sludge WAS). The second digester (methaniser) was fed by the residual effluent received from the hydrogeniser. The methaniser was operated by correcting to optimise conditions of pH to 6.5 ± 0.3 and an HRT of 10 days and sub-optimal feed conditions of C:N 18 and OLR of 1.9 g VS/L/d. Acclimatised seed inoculum was developed in two lab-scale mesophilic CSTRs, one for hydrogen production and one for methane. The hydrogeniser was operated at a temperature of $37^{\circ}\text{C}\pm 0.2$ and an HRT of 5 days. A co-blended waste of IFW and SS with OLR of 6 g VS/L/d was continuously supplied to the reactor. The conditions were: pH, 5.5 ± 0.3 ; alkalinity, 5,000 mg/l; VFA, 46,200 mg/L and hydrogen composition, 38.5%. The methaniser was operated at a temperature of $37^{\circ}\text{C}\pm 0.2$ and an HRT of 10 days and feed with a co-blended waste of IFW and SS with OLR of 3.4 g VS/L/d. The conditions were: pH, 6.5 ± 0.3 ; alkalinity, 5,000 mg/L; VFA, 31,200 mg/L and methane composition, 70.1%.

2.2 Experimental setup

A phase separated two-stage hydrogeniser followed by methaniser was operated in semi-continuous mode (feeding once a day). Both digesters were fabricated using 0.8cm thick perspex with a gas-tight, hermetically sealed, rubber sealing, the internal temperature ($37\pm 2^{\circ}\text{C}$) of the contents was controlled by a thermostatically heat controlled jacket. The total volume of the hydrogen producing digester was 1.67 L with a working volume 1.5 L (Figure 1) and the total volume of the methaniser was 5.0 L with a working volume 4.0 L (Figure 1). Feeding of the hydrogeniser was carried out using the top feed inlet; the second

digester (methaniser) was fed by residual effluent received directly from the hydrogeniser effluent port, fixed at effective height. The gas was continuously collected from the gas ports and it was volumetrically measured at normalized pressure using a water displacement method; later the gas volume was then corrected to STP. Both digesters were mechanically mixed at 100 rpm with a paddle guided by an IKA overhead stirrer at 100 rpm and a cycle: reaction time of 20 minutes with mixing every hour, up to 16 hours followed by continuous mixing for 1 hour and settling of 6 hours. Manual correction of pH to a value of 5.5 for the hydrogeniser and 6.5 for the methaniser was undertaken using 6M NaOH and 1M HCl.

2.3 Analytical methods

The analytical parameters of total solids (TS), volatile solids (VS), total alkalinity (TA), ammoniacal nitrogen ($\text{NH}_4\text{-N}$) and total volatile fatty acids (TVFA) were carried out following the procedures outlined in APHA (1998 [1]). Samples for analysis of TA, $\text{NH}_4\text{-N}$, and TVFA were centrifuged at 60,000 rpm for 1 hour prior to analysis. The elemental composition of all feed-stocks was determined with vanadium pentoxide by flash combustion method using the CHNSO Analyser (Thermo Flash EA-1112 series, Italy). Gas composition was analysed using a gas chromatograph (Hewlett Packard HP 5890 Series II, USA) with a thermal conductivity detector TCD using a PLOT capillary molisieve column (30 m \times 0.32 mm \times 12 μm ; Hewlett Packard HP, USA). The temperature of injector, detector and column were kept at 80, 90 and 40°C respectively. To determine hydrogen and methane, nitrogen was used as carrier gas with a flow of 6 mL/min. VFA profile (organic acids) were quantified using the same GC and flame ionization detector FID using a wall coated open tubular (WCOT) capillary fused silica column (25 m \times 0.32 mm \times 0.44 mm; Nordion, FI). Before analysing the acids and alcohols, samples were acidified with 10% and 3% formic acid. The temperature of injector, detector and column were kept at 200, 185 and 160°C respectively. Helium was used a carrier gas with a flow of 60 mL/min.

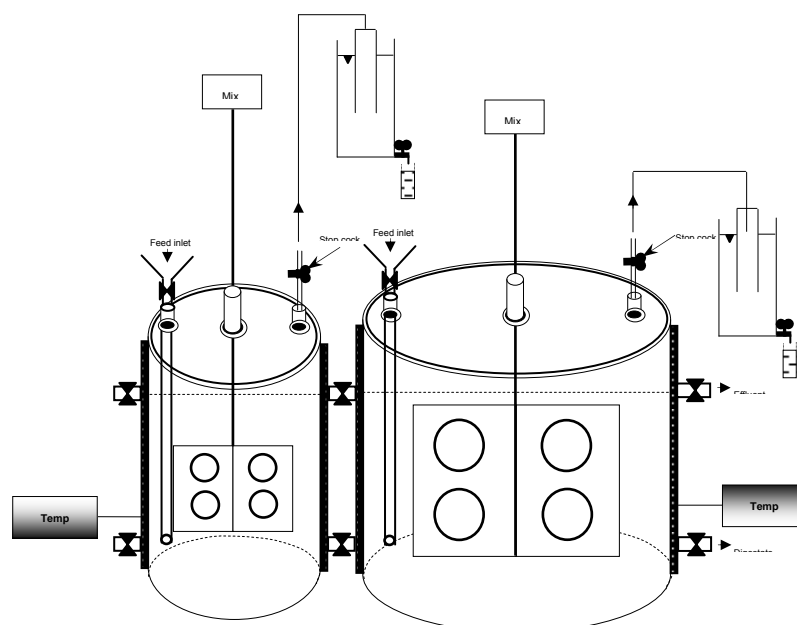


Figure 1: Laboratory scale two-stage phase-separated hydrogeniser followed by methaniser.

3 Results and Discussion

3.1 Recovery of hydrogen and methane using the two-stage phase-separated process

The specific hydrogen production (SHP) was observed in the range of 0.49 to 0.54 L with an average of 0.53 L (Table 1). The SMP of the methaniser of the second stage was observed in range of 2.2 to 3.7 L with an average of 3.2 L. An improvement in SHPR was observed and found in range of 0.3 to 0.4 L/(L.d) (rec vol) with an average of 0.4 L/(L.d). The SHPR confirmed the earlier SHPR from the single-stage CTSR (HRT of 5 days) (Siddiqui, 2010 [2]). A similar gradual improvement was observed in SMPR and varied over the range of 0.6 to 1 L with an average of 0.8 L/(L.d) (Table 1). A decrease of 0.4 L/(L.d) was noted compared to the average SMPR of CTSR (HRT of 10 days) (Siddiqui, 2010 [2]). It was thought that the reduction in SMPR was due to a decrease in the OLR from 3.3 g VS/L/d (Table 1) to 1.6 g VS/L/d (Table 1). Nevertheless the SMPR was twice the SHPR. This verified that the volumetric methane production is more than the volumetric hydrogen production. The concentration of hydrogen (38%) was comparable to the earlier study; in contrast a decrease in methane level was observed in the second stage.

The efficiency of anaerobic fermentation is determined mainly through production of intermediary metabolites and the biohydrogen yield from a given substrate. The major organic acids produced were acetic acid and butyric acid and the production of higher organic acids (acetic acid and butyric) produced at shorter retention times showed that hydrolysis of substrate would give an indication of the maximum biohydrogen yield. The hydrogen yield varied in the range of 106.1 to 146.7 mL/g VS_{removed} (Table 1) with average of 129.1 mL/g VS_{removed} (Table 1). The hydrogen yield was very similar to earlier yields achieved during single stage CSTR of HRT of 5 days (Siddiqui, 2010 [2]). It was observed that the effluent from the hydrogeniser had a residual volatile solids and organics acids that was amenable for recovery of biomethane in the subsequent methaniser. Using the same residual effluent, the methane yield varied in range of 496 to 762.3 mL/g VS_{removed} (Table 1) with average yield of 617.6 mL/g VS_{removed} (Table 1). A significant improvement in methane yield was noted compared to the earlier, single-stage CTSR 476 mL/g VS_{removed} (Siddiqui, 2010 [2]) and was 1.3 fold higher. This enabled the conclusion that installing a hydrogeniser, hydrolysed the organic acids and was ideal for increasing the methane yield. The methane yield achieved was greater than other authors observed, for single and two stage instance 0.133 to 0.638 m³/kg VS.

Table 1: Performance of phase-separated two-stage reactors and effluent characteristics after hydrogen and methane recovery.

Properties	Unit	2 stage digester	
		Hydrogeniser	Methaniser
HRT	d	5	10
VS _{removed} Max(Avg)	%	56.5(47)	72.7(80.7)
SHP or SMP Max(Avg)	L	0.57(0.53)	3.7(3.2)
H ₂ or CH ₄ yield Max(Avg)	mL/g VS _{removed}	146.7(129.1)	617.6(476)
H ₂ or CH ₄ Avg	%	38	70
SHPR or SMPR Max(Avg)	L/(L.d)	0.4(0.4)	0.93(0.8)
Effluent pH ^a	-	5.3	7.2
Effluent C:N	-	18	-
Effluent NH ₄ -N ^a	mg/L	33.2	42
Effluent TVFA ^a	mg/L	38,173	27,828
Effluent Magnesium ^b	mg/L	199.7	140
Effluent Phosphate ^b	mg/L	121.9	12.7
Effluent Alkalinity ^a	mg/L	4,750	5,833
Effluent Struvite ^b	mg/L	2,597	814

^a based on average results of effluent of hydrogeniser and methaniser

^b based on mixing the last three samples attained during the operation of the reactors

3.2 Effluent characteristics

The residual ammonium and alkalinity levels were low but pH was still noted above neutral range (Table 1). This might be due to the efficient operational control of the two-stage reactor that has separated the hydrolysis step (as hydrogenesis) and methanogenesis regardless of pH (4.6) of the ideal blend (Siddiqui, 2010 [2]). In order to take advantage of the effluent characteristics: neutral pH, low level of ammonium nitrogen and high levels of phosphate and magnesium (Table 1), a trial was run to monitor the theoretical potential of struvite (Mg NH₄ PO₄ · 6H₂O) recovery. This was established as 2,597 mg/L in the effluent from hydrogeniser and 814 mg/L in effluent from the methaniser. The theoretical struvite potential demonstrated the feasibility of nutrient precipitation by reducing the tertiary treatment process. The experimental efficiency of struvite recovery was 70%. In a similar way, the residual organic acid level became steady and major organic acid produced were acetic acid and ethanol. Initially a high level of acetic acid (55% v/v) was observed that reduced to (38% v/v). In contrast an increase in ethanol production from 30% to 53% (v/v) was observed during the two stage process.

3.3 Total efficiency of two-stage phase-separated reactor

The efficiency of the anaerobic digestion process was considered by analysing the amount of volatile solids destroyed and total energy produced. The independent VS destroyed efficiency of the hydrogeniser was recorded as 47% (Table 1) and this increased to (80.7%)

for two stage process (Table 1). The performance efficiency of the two-stage process was significantly higher comparing to single stage CSTR (HRT of 15 days) 66.4% (Siddiqui, 2010 [2]) and also the efficiency was higher compare to ealier work.

The total energy production of two-stage phase-separated hydrogen and methane production was high compare to single-stage methaniser only. The hydrogen and methane yield was noted as 129.1 and 476 mL/g VS_{removed} (Table 1). Significant improvement in methane yield was noted compare to the earlier single-stage CTSR operated at HRT of 10 and 15 days. The energy potential of optimum condition in single stage hydorgeniser is 2.27 MW/tonne VS_{fed}. Using the two phase and sub optimal conditions improves the energy potential to 8.27 MW/tonne VS_{fed}.

4 Conclusions

- A two-phase reactor was operated with the first phase at pH 5.5 and OLR of 6 g VS/L/d. A hydrogen yield of 129.1 mL/g VS_{removed} was obtained with a VS destruction of 27%
- The digestate was close to optimal for the second phase which operated at a pH of 6.5 and an OLR of 1.6 g VS/L/d to give methane yield of 476 mL/g VS_{removed}
- The two-phase reactor had a combined energy yield of 8.27 MW/tonnes of VS_{fed} and a VS destruction of 80.7%
- The effluent characteristics showed a viability of struvite, acid and ethanol recovery. The theoretical residual concentration of struvite from the hydrogeniser and methaniser were 2,597 mg/L and 814 mg/L. The organic acid produced were acetic acid (38% vol basis) and ethanol (53% vol basis).

References

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