Biogas Production from Anaerobic Co-digestion of Food Waste with Dairy Manure in a Two-Phase Digestion System

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Abstract Co-digestion of food waste and dairy manure in a two-phase digestion system was conducted in laboratory scale. Four influents of R0, R1, R2, and R3 were tested, which were made by mixing food waste with dairy manure at different ratios of 0:1, 1:1, 3:1, and 6:1, respectively. For each influent, three runs of experiments were performed with the same overall hydraulic retention time (HRT) of 13 days but different HRT for acidification (1, 2, and 3 days) and methanogenesis (12, 11, and 10 days) in two-phase digesters. The results showed that the gas production rate (GPR) of co-digestion of food waste with dairy manure was enhanced by 0.8-5.5 times as compared to the digestion with dairy manure alone. Appropriate HRT for acidification was mainly determined by the biodegradability of the substrate digested. Three-, 2-, and 1-day HRT for acidification were found to be optimal for the digestion of R0, R1, and R2/R3, respectively, when overall HRT of 13 days was used. The highest GPR of 3.97 L/L·day was achieved for R3(6:1) in Run 1 (1+12 days), therefore, the mixing ratio of 6:1 and HRT of 1 day for acidification were considered to be the optimal ones and thus recommended for co-digestion of food waste and dairy manure. There were close correlations between degradation of organic matters and GPR. The highest VS removal rate was achieved at the same HRT for acidification and mixing ratio of food waste and dairy manure as GPR in the co-digestion. The two-phase digestion system showed good stability, which was mainly attributed to the strong buffering capacity with two-phase system and the high alkalinity from dairy manure when co-digested with food waste.

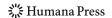
Keywords Anaerobic co-digestion \cdot Biogas \cdot Dairy manure \cdot Food waste \cdot Mixing ratio \cdot Two-phase digestion system

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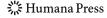
Introduction

In 2006, approximately 251 million tons of municipal solid waste (MSW) is generated in the USA [1]. Except recyclable materials such as paper, plastics, etc., food waste is the largest waste stream and accounts for 12.5% of total MSW. Food waste includes food residues, fruits, vegetables, grain, and milk, etc. from retails, restaurants, and households. Due to the characteristics of food waste with 70–90% moisture content and high organic content of volatile solid (VS) [2, 3], improper treatment would cause serious environmental problems, such as odor and leachate production [4].

Landfill is one of conventional approaches for food waste disposal; however, it would be limited in the near future, due to the concerns of greenhouse gas emission or other negative impacts on the environment. In Europe, the regulations have been set to strictly limit the disposal of organic waste by landfill and the landfill fee has been raised as well [5]. Anaerobic digestion has been recognized as environmentally friendly technology to convert organic solid waste such as animal manure, food waste, and organic fraction of MSW into renewable energy in biogas form [6]. However, digestion process tends to fail, when one readily degradable organic matter is used as sole substrate without external nutrients and buffering agent [7]. Co-digestion with other wastes could provide more suitable physicochemical property of feedstock and more balanced nutrients for efficient digestion and high biogas production; thus, it could be used to solve the problem or help achieve higher digestion efficiency.

There are a number of wastes, which could be used to co-digest with food waste. Dairy manure is a better one for co-digestion due to the availability and its suitable physicochemical characteristics. Dairy manure is an important organic waste from dairy industry with the estimated annual production of 42 million tons on dry basis in the USA [8]. As increasingly stringent requirements have been set to limit the direct land application, anaerobic digestion for bioenergy production would be one of options. On the other hand, dairy manure is an ideal base feedstock for co-digestion with other wastes due to its high buffer capacity, which is critical for the anaerobic digester achieving stable performance [7]. Several studies have been made on the co-digestion of various organic wastes with dairy manure. Callaghan et al. [9] investigated the co-digestion of cattle slurry with other organic wastes such as fruit and vegetable waste, and found that co-digestion could obtain more methane production than the digestion with cattle manure alone. El-Mashad and Zhang [10] evaluated the performance of the co-digestion of food waste with dairy manure using a completely mixed reactor, and obtained a high biogas yield of 504 mL/g VS at the organic loading rate (OLR) of 2-4 g VS/L·day in semi-continuous operation.

Besides feedstock used for co-digestion, digestion system is another important factor. For the highly biodegradable waste, two-phase digestion system was believed to be more advisable [6]. In two-phase digestion system, the digestion process is separated into an acidification phase and a methanogenesis phase, which could provide more suitable condition for acid-forming bacteria and methane-forming bacteria respectively, thus enhance the overall activity in digester. A number of studies presented the advantages of two-phase system in the anaerobic digestion of organic wastes [6, 11, 12]. Bouallagui et al. [11] reported that two-phase anaerobic digestion of fruit and vegetable wastes using two coupled anaerobic sequential batch reactors resulted in high process stability, significant biogas productivity, and better effluent quality. Dinsdale et al. [12] investigated two-phase anaerobic digestion of fruit/vegetable and waste activated sludge, and achieved stable performance at an overall OLR of 5.7 g VS/L·day using inclined tubular digester.



In this study, dairy manure was used to co-digest with food waste in a two-phase digestion system. For co-digestion, the performance is determined by a number of factors, but the composition of influent or the mixing ratio is believed to be a critical one [13]. In the study of Lehtomäki et al. [14], the highest specific methane yield was obtained with 30\% crop and 70\% cow manure in the feedstock. However, the improper mixing ratio of influent in the co-digestion would cause poor performance of digester. Gelegenis et al. [15] reported that the digester became unstable at whey fraction in excess of 50% in the codigestion of whey with diluted poultry manure. For two-phase digestion, total hydraulic retention time (HRT) and the HRT for acidification and methanogenesis phase is important. Ghosh [16] suggested that an HRT of 3 days is needed for activated sludge achieving the optimum acidogenic fermentation in the two-phase anaerobic digestion. Banerjee et al. [17] stated that the increase in HRT from 18 to 30 h could improve the extent of acidification and substrate solubilization when starch wastewater was co-digested with primary sludge by bench digester. However, the influence of longer acidification HRT and high level of acidification on the biogas production is disputable. Lettinga and Hulshoff Plo [18] recommended partial pre-acidification and short HRT varying from 6 to 24 h for soluble non-complex wastewaters and complex partial soluble wastewater. For the methanogenesis, 10 days of HRT was reported to be the optimal one for FW in terms of biogas production by Kim et al. [19]. Dinsdale et al. [12] investigated the two-stage digestion of fruit/vegetable waste and waste-activated sludge, and found that increasing the methanogenic HRT from 10 to 13 days could decrease the effluent volatile fatty acids (VFA). It can be seen from above studies that for the different wastes to be co-digested in two-phase system, the appropriate mixing ratio and HRT would be different. Little information is available for the co-digestion of food waste and dairy manure in two-phase system. Therefore, specific research needs to be conducted.

The objectives of the present work were to investigate the performance of co-digestion of food waste with dairy manure in a two-phase system and determine appropriate co-digestion parameters in terms of biogas production, organic matter degradation, and digestion system stability.

Materials and Methods

Materials Preparation

Food waste was collected from Regency Dinning Center at Washington State University (WSU) at Pullman, which was composed of fruits, vegetables, starch, and grease, etc. The food waste was shredded into 5-10-mm size pieces by food grinder. Dairy manure was collected from WSU dairy center. After dilution with tap water, the dairy manure was separated by a screen to remove fibers. The prepared food waste and dairy manure materials were stored in a freezer at -20 °C for later uses.

The characteristics of dairy manure and food waste are represented in Table 1. As compared to dairy manure, food waste contained higher percentages of total solids (TS), VS, chemical oxygen demand (COD), VFA, protein, lipids, total ammonia nitrogen (TAN), and total Kjehldahl nitrogen (TKN), but lower pH, alkalinity, carbohydrate, and free ammonia (FA). Dairy manure had stronger buffer capacity as indicated by higher alkalinity of 2.68 g CaCO₃/L.

To investigate the effect of influent characteristics on co-digestion performance, four influents of R0, R1, R2, and R3 were made by mixing food waste with dairy manure at

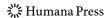


Table 1 Characteristics of food waste and dairy manure.		Food waste	Dairy manure		
	TS, g/L	291±0.8	20.7±0		
	VS, g/L	260 ± 0.1	15.3 ± 0		
	COD, g/L	368.4 ± 30.6	23.6 ± 1.8		
Data are presented as the mean± SD of three replicates a Carbohydrate, protein, and lipids percentages were based on the VS of food waste and dairy manure and carbohydrate was determined by the total VS	VFA, g/L	8.2 ± 1.8	2.4 ± 0.7		
	рН	4.2 ± 0.2	7.2 ± 0.1		
	Alkalinity, g/L	0	2.7 ± 0.4		
	Carbohydrate ^a , %	59±4.3	71.4 ± 2.8		
	Protein ^a , %	33 ± 3.2	26.6 ± 2.4		
	Lipids ^a , %	8 ± 1.1	2 ± 0.4		
	TAN, g-N/L	1.3 ± 0.1	0.3 ± 0		
	FA, mg-N/L	0.02 ± 0	4.2 ± 0.5		
deducting protein and lipids in food waste and dairy manure	TKN, g-N/L	15.4 ± 1.5	1.0 ± 0.1		

different ratios (mixing ratio of FD) of 0:1, 1:1, 3:1, and 6:1 on VS basis, respectively. The corresponding food waste wet fraction, TS, VS, COD, TAN, and FA were calculated. The characteristics of the influents used are shown in Table 2. It is distinct that the TS, VS, and COD increased significantly with the increase of mixing ratio of FD, while the TAN and FA of four influents were on the same level.

Two-Phase Digester

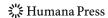
Two completely mixed reactors were used as the acidific and methanogenic reactors. The volume of the second-phase reactor for methanogenesis was 3.5 L with a working volume of 2.0 L, while the volume of the first-phase reactor for acidification varied with HRT used. The effluent of the first-phase reactor was fed into the second-phase as influent once a day. The digestion system was thus performed in semi-continuous mode. The digesters were seeded using the anaerobic sludge taken from a manure digester in operation near campus. The digesters were operated at mesophilic temperature $(35\pm2~^{\circ}\text{C})$.

Experimental Design

For each influent, three runs of experiments were operated with the same overall HRT but different HRT for acidification and methanogenesis. The overall 13 days of HRT; 1, 2, and 3 days of HRT for acidification; and 12, 11, and 10 days of HRT for methanogenesis were

Table 2 Characteristics of four influents made by mixing food waste with dairy manure (FD).

Influents	R0	R1	R2	R3
Mixing ratio of FD on VS basis	0: 1	1: 1	3: 1	6: 1
Food waste wet fraction, %	0	5.6	15.0	25.7
TS, g/L	20.7 ± 0	35.8 ± 0.1	61.3 ± 0.1	90.2 ± 0.2
VS, g/L	15.3 ± 0	29.0 ± 0	52.0 ± 0	78.2 ± 0
COD, g/L	23.6 ± 1.8	42.9 ± 3.4	75.3 ± 6.1	112.2 ± 9.2
TAN, g-N/L	0.25 ± 0.03	0.31 ± 0.03	0.4 ± 0.03	0.51 ± 0.04
FA, mg-N/L	4.2 ± 0.5	4.0 ± 0	3.6 ± 0	3.1 ± 0



used in this study according to the results from Ghosh [16] and Kim et al. [19]. The different HRT design for acidification and methanogenesis was aimed to investigate the effect of different HRT on the performance of a two-phase digestion system. The overall experiment design is shown in Table 3. For Run 1, the HRT for acidification and methanogenesis was 1+12 days while 2+11 days for Run 2 and 3+10 days for Run 3.

Methods

TS, VS, pH, and alkalinity were tested according to the standard methods [20]. COD was analyzed by COD analyzer (HACH, DR 2500, USA). The content of lipids was determined by Soxhlet method with petroleum ether as eluent. TAN and TKN were analyzed by TKN analyzer (Foss 2003, Sweden). CH₄ and CO₂ compositions were determined through a Varian GC (Palo Alto, CA, USA) equipped with a thermal conductivity detector. VFA was measured with a high-performance liquid chromatography (HPLC) (Varian, Walnut Creek, CA, USA) with an organic acid column of Aminex HPX-87H (BioRad, USA). The temperature of the column was 65 °C, and 0.005 N of sulfuric acid was used as mobile phase with a flow rate of 0.55 mL/min. The peak for the UV-absorption spectroscopy was achieved at wave length of 210 nm and the resulted chromatogram was integrated and plotted using Star chromatographic software. The samples for VFA analyses were first centrifuged at 10,000 rpm for 5 min, the supernatants were then filtered through a 0.45 μ m filter, and filtrates were collected in HPLC sample vials for analyses.

Results and Discussions

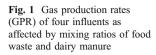
Biogas Production

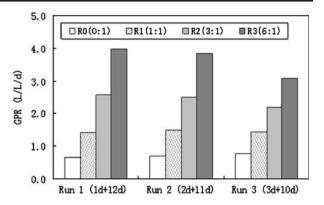
After the digestion process reached steady state, the daily biogas production was recorded. The gas production rate (GPR), which was based on the working volume of digester, is shown in Fig. 1. For each Run, GPR showed the same trend, which significantly increased as the mixing ratios of FD changed from 0:1 to 1:1, 3:1, and 6:1. The GPRs of R1, R2, and R3 were 1.42–1.48, 2.18–2.56, and 3.09–3.97 L/L·day, which were 0.8–1.1, 1.8–3.6, and 3.0–5.5 times greater than that of R0. This was mainly due to the increased amounts of organic matters loaded, as indicated by OLRs of 1.2, 2.2, 4.0, and 6.1 g VS/L day in R0, R1, R2, and R3, respectively. It was found that within the range of OLRs applied; the GPR kept increasing with OLR loaded, implying that the digestion system might be capable of using even higher OLR. A linear regression equation was established as shown in Fig. 2. It can be observed that GPR was positively correlated with OLR applied as indicated by

 Table 3 Experimental design of the two-phase digestion system.

Influents	Mixing ratio of FD	OLR (g VS/L·day)	HRT in R	un 1	HRT in R	un 2	HRT in Run 3		
			1st phase (day)	2nd phase (day)	1st phase (day)	2nd phase (day)	1st phase (day)	2nd phase (day)	
R0	0:1	1.2	1	12	2	11	3	10	
R1	1:1	2.2	1	12	2	11	3	10	
R2	3:1	4.0	1	12	2	11	3	10	
R3	6:1	6.1	1	12	2	11	3	10	





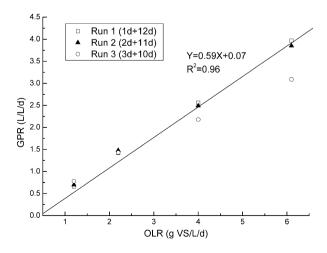


correlation coefficient (R^2 =0.96). The highest GPR was obtained at R3(6:1) for all three Runs; therefore, the mixing ratio of FD of 6:1 was considered to be the optimal mixing ratio and thus recommended for co-digestion of food waste and dairy manure.

For each mixing ratio of FD, three runs of experiments were conducted using three different HRT combinations for acidification and methanogenesis. The effect of HRT for acidification and methanogenesis on the performance of the digestion system was evaluated in terms of GPR. The GPR for each mixing ratio of FD at different HRT combinations is shown in Fig. 3.

For the digestion with dairy manure alone (R0), the highest GPR of 0.78 L/L·day was obtained in Run 3 (3+10 days), which was 17.5% higher than that in Run 1 (1+12 days). Dairy manure contains certain amount of relatively hard degradable components such as cellulose and lignin, thus needs longer time for hydrolysis in the first phase. As it is known that hydrolysis is the rate-limiting step in the anaerobic digestion of organic solids [21]. The longer HRT for acidification in Run 3 would allow complete hydrolysis of organic solids and provide more acidic compositions for methanogenesis in the second-phase, thus, enhance biogas production. For the digestion of R1(1:1), the highest GPR of 1.48 L/L·day was achieved in Run 2 (2+11 days). However, for the digestion of R2(3:1) and R3(6:1),

Fig. 2 Gas production rates (GPR) of four influents as affected by organic loading rates (OLR)



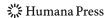
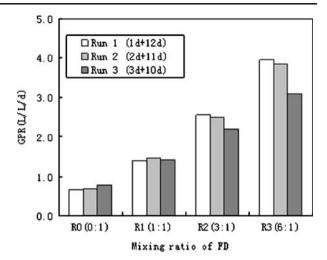


Fig. 3 Gas production rates (GPR) of four influents as affected by HRTs



both obtained the highest GPR (2.56 and 3.97 L/L·day) in Run 1 (1+12 days). The results clearly showed that HRT for acidification is an important factor impacting the biogas production. As food waste proportion in the influent increased, the optimal HRT for acidification was decreased accordingly. Shorter HRT of 1 day for acidification was required for the influents of R2 and R3, while longer one of 3 days for acidification was needed for the influent of R0. This was mainly due to the higher percentage of relatively readily biodegradable compounds contained in the influents of R2 and R3, which took less time for hydrolysis and acidification. Therefore, 3, 2, and 1 day HRT for acidification could be recommended for the co-digestion of R0, R1, R2/R3 in the two-phase system, respectively and corresponding HRT for methanogenesis would be 10, 11, and 12 days, respectively. The result also suggests that for two-phase digestion, HRT required for acidification is mainly dependent on biodegradability of feedstock. Shorter HRT for acidification and longer HRT for methanogenesis would be desired for readily biodegradable feedstock. However, optimal HRT for acidification needs to be determined by specific study, when different feedstock is applied.

Methane contents for all runs were analyzed during the digestion experiments. They showed very similar trends, thus, only the methane contents of four influents digested in Run 1 are presented (Fig. 4). It was found that the average methane content for R0 was 59.0%, while 62.2%, 62.1%, and 62.8% for R1, R2, and R3, respectively. Statistical analysis showed that there was no significant difference in methane contents among the four influents at different mixing ratios of FD (ANOVA one-way, F=0.001, P>0.05). Therefore, more attention should be focused on GPR as discussed above.

Degradation of Organic Matters

Biogas is produced through the conversion of organic matters by anaerobic microorganisms. With the conversion of organic matters into biogas, the amount of organic dry matters would be reduced accordingly. Based on mass balance, the removal rates of TS and VS were calculated and the results are shown in Fig. 5.

It was observed that the removal rates of TS and VS were affected by mixing ratios of FD and HRT obviously. For the digestion of R2(3:1) and R3(6:1), VS removal rates

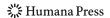


Fig. 4 Methane content of four influents at different mixing ratios of food waste and dairy manure in Run 1. Data are presented as the mean of triplicates and the *error bars* show the standard deviation

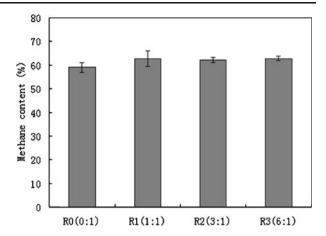
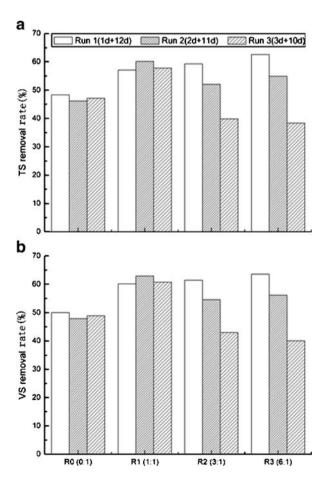
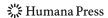


Fig. 5 Removal rates of TS (a) and VS (b) as affected by HRT and the mixing ratios of food waste and dairy manure

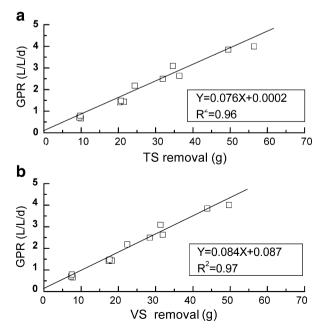




decreased as HRT for acidification increased. Both obtained the highest VS removal rates of 61.4% and 63.6% at shorter HRT of 1 day in Run 1(1+12 days). It is evident that HRT for acidification is an important factor impacting biogas production as well as the degradation of organic matters. It is found that the changes of VS removal rate well corresponded to that of GPR as discussed above; the highest VS removal rate was achieved almost at the same HRT for acidification and mixing ratio of FD as GPR, showing the close relationship between degradation of organic matters and biogas production. For the digestion of R0(0:1) and R1(1:1), both obtained similar VS removal rates in three runs, 47.9%–50.1 and 60.1%–62.9, respectively. It might due to the OLR applied for R0 and R1 were relatively low, thus, the difference in VS removal rates among three runs was not as significant as those of R2 and R3. The trends of TS removal rates were very similar to that of VS, and the differences were the values of removal rates. Comparing Fig. 6a and b, it can be seen that the values of removal rates of TS were generally lower than VS.

To determine the relationship between GPRs and the removal rates of TS and VS, GPR was plotted against the removal amounts of TS and VS, respectively. Three linear regression equations were established based on the data obtained in this study (Fig. 6). It can be seen that there were significant correlations between GPR and the removal amount of TS and VS, which were indicated by high correlation coefficient of R^2 . Generally speaking, GPR would increase with the increase of TS and VS removal rate. This implies that more organic matters need to be converted in order to obtain more biogas production. The equations could also be used to predict biogas production when either TS or VS removal rate is known.

Fig. 6 Gas production rates (GPR) as affected by the TS removal (**a**) and VS removal (**b**)



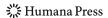
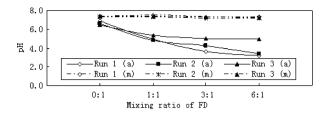


Fig. 7 pH in the acidification (a) and the methanogenesis (m) phase



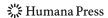
Process Stability

It is important for a digester to operate in stable state with good performance. The stability of one digestion system depends on a number of factors, of which are pH, VFA, TAN, FA, and alkalinity. These parameters were measured in this study in order to evaluate the stability of the two-phase system for co-digestion of food waste and dairy manure. The results are shown in Fig. 7 and Table 4.

In acidification phase, the VFA concentrations for the higher mixing ratios of FD with R2 and R3 in Run 2 and Run 3 generally maintained at higher levels, while at lower levels for the lower mixing ratios of FD with R0 and R1 in three Runs. This might be explained by two facts: (1) higher mixing ratios of FD with R2 and R3 brought more VFA from influents into the digesters and (2) longer HRT for acidification with Run 2 and Run 3 allowed more substrate to hydrolyze and produce more VFA. Compared to acidification phase, the VFA concentrations in methanogenesis phases were significantly lower. It was found that the VFA concentrations of R2 and R3 in Run 3 were 3.70 and 4.14 g/L. Both values are obviously higher than 1.5 g/L, which is commonly considered to be the limit for allowing stable operation of one biogas digester [22]. However, this two-phase system still maintained in normal operation at such high VFA concentrations. This could be attributed to the strong buffering capacity with two-phase system as well as the high alkalinity of above 3.1 g CaCO₃/L with co-digestion in the digesters. TAN and FA as the potential inhibitors to methanogens in this study were lower than the reported inhibition level [23], respectively, indicating that TAN and FA are not major concerns for the system stability in this case. The stability of the digestion system could be further verified by the changes of pH values during digestion process as shown in Fig. 7. It can be seen that although the pH

Table 4 VFA, TAN, FA, and alkalinity in the effluents.

Influents	Run 1 (1+12 days)				Run 2 (2+11 days)			Run 3 (3+10 days)				
	R0	R1	R2	R3	R0	R1	R2	R3	R0	R1	R2	R3
VFA in acidification, g/L	1.56	3.41	4.02	3.83	2.52	4.88	6.08	6.92	2.45	4.59	7.39	8.38
VFA in methanogenesis, g/L	0.07	0.03	3.01	3.04	0	0.03	2.96	3.09	0.25	1.25	3.70	4.14
TAN, g-N/L	0.4	0.9	1.6	2.1	0.4	0.8	1.4	1.9	0.4	0.9	1.8	1.9
FA, mg-N/L	10.6	33.5	37.3	44.3	8.9	18.5	23.4	32.7	10.2	24.0	41.6	33.3
Alkalinity, g CaCO ₃ /L	3.6	5.1	6.6	7.5	3.7	4.8	6.4	7.8	3.1	5.0	7.1	7.5



in the acidification phase fell down as low as 3.2, the pH in methanogenesis was still able to maintain between 7.2–7.4, which are in the range for normal operation of anaerobic digestion process.

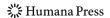
Conclusions

The GPR of co-digestion of food waste and dairy manure was enhanced by 0.8–5.5 times as compared to the digestion with dairy manure alone. Appropriate HRT for acidification was mainly determined by the biodegradability of the substrate digested, which was greatly affected by the mixing ratio of FD in this study. Three, 2, and 1 day HRT for acidification were found to be optimal for the digestion of R0, R1, and R2/R3, respectively, when overall HRT of 13 days was used. The highest GPR of 3.97 L/L·days was achieved for R3(6:1) in Run 1 (1+12 days), therefore, the mixing ratio of 6:1 and HRT of 1 day for acidification were considered to be the optimal ones and thus recommended for the co-digestion of food waste and dairy manure. There were close correlations between degradation of organic matters and biogas production, the change of VS removal well corresponded to that of GPR. The highest VS removal rate was achieved at the same HRT for acidification and mixing ratio of FD as GPR in the co-digestion. The two-phase digestion system showed good stability, which was mainly attributed to the strong buffering capacity with two-phase system and the high alkalinity from dairy manure when co-digested with food waste.

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