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Synergistic effect and biodegradation kinetics of sewage sludge and food waste mesophilic anaerobic co-digestion and the underlying stimulation mechanisms

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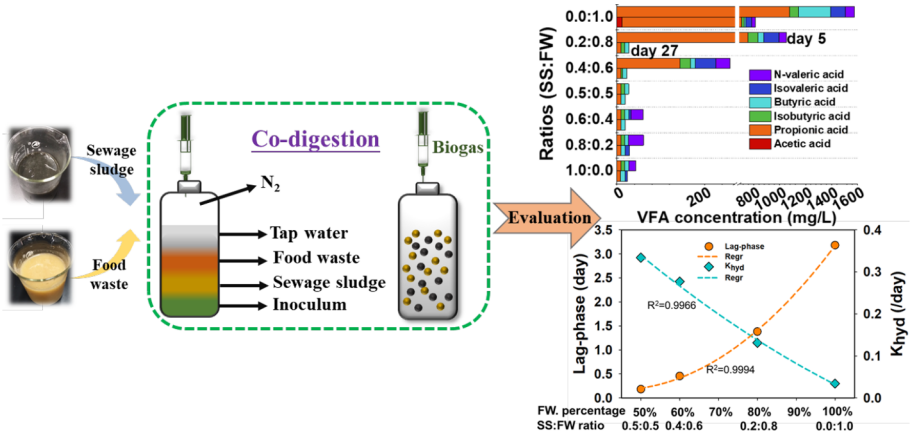
GRAPHICAL ABSTRACT



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ABSTRACT

Anaerobic co-digestion has attracted much attention due to its unique advantages compared with the anaerobic mono-digestion. In this work, the synergistic effect and biodegradation kinetics of sewage sludge (SS) and food waste (FW) were studied during anaerobic co-digestion. Biochemical methane potential (BMP) assay and three kinetic models were used to detect and analyze methane production potential during co-digestion under different ratios of SS and FW. The SS:FW ratios of 0.5:0.5 showed very high methane recovery, with the methane productivity increasing by 4.59 times (50.30 ± 10.37 mL/g-VS/day), the lag-phase shortening by 11.53 times (0.182 day) and the hydrolysis rate increasing by 3.88 times (0.334/day) compared with the SS mono-digestion. Moreover, correlation model parameter analysis indicated the Cone model has the best fitness to the

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experimental data. These results confirmed the superiority of co-digestion and the existence of synergy, which can greatly accelerate the biodegradation process and more effectively recover bioenergy.

1. Introduction

During the wastewater treatment process, the production of sewage sludge (SS) is unavoidable. In recent years, the rapid industrialization and urbanization has caused the increasing quantity of wastewater, which leads to an increase in sludge production [1,2]. For instance, the sludge production reached up to nearly 30 million dry metric tons in China in 2015 [3], and it will be over 80 million tons in 2020. Deficiently, less than 20% of the total produced sludge attains proper treatment [4]. Due to the presence of many kinds of hazardous materials (some organic contaminants, pathogens, heavy metal, etc.) [5], the un-stabilized sludge could pollute the environment and cause the waste of resources. Therefore, the stabilization and decontamination of SS is a hot topic for industry and academia.

Anaerobic digestion (AD), as an effective and well-proven technology for the disposal of SS, achieves triple benefits such as the stabilization, the energy recovery (mainly methane), and the protection of environment [6]. Nevertheless, the process of mono-digestion of sludge tends to be slow and unstable due to nutrition deficiency, low organic loading rate [7], low biodegradability [8], and high toxicity of contaminants (fragrances, antibiotics, etc.) [9]. The co-digestion of two or more substrates can properly resolve such problems by adjusting the unbalanced substance composition, which is able to enhance the buffering capacity, accelerate the hydrolysis rate and thus improving the stability of system, and biogas production [10–12]. The co-digestion possesses better energy recovery efficiency, thereby attracting the more attention from researchers. Solé-Bundó et al. [13] co-digested SS and microalgae and confirmed that co-digestion boosted the stabilization process and digestate dewaterability. Fountoulakis et al. [14] mixed the SS with 1% glycerol (volume ratio), and found that the methane production rate reached 2353 ± 94 mL/day, nearly doubling that of SS mono-digestion. Besides, the improved performance was also observed during the co-digestion of SS with coffee waste [15], swine and poultry manure [16] and agricultural wastes [17].

Food waste (FW) is a typical biomass, which generates in large amounts during the living and production process. The high biodegradability of FW makes it a desirable co-substrate to co-digest with SS. Dai et al. [18] found that increasing the proportion of FW improved the biogas production and volatile solids (VS) reduction during the co-digestion of dewatered sludge (DS) and FW. Prabhu and Mutnuri [19] reported the maximum biogas of 823 mL/g-VS at the ratio of SS and FW of 1.0:2.0. Although several studies have demonstrated the great performance of the co-digestion of SS and FW, the synergies and biodegradation kinetics during the co-digestion are still not elaborated and recognized clearly [20]. In addition, using one model to describe the biodegradation kinetics might be not accurate and scientific enough due to the complexity of the anaerobic process. In our previous research, Zhen et al. [6] used three models (i.e. first-order kinetic, modified Gompertz, and Cone models) to explore the biodegradation kinetics of the microalgae and FW co-digestion and found that different models have different precisions for the same experimental data. Hence, the description of biodegradation kinetics is more reliable and truer if several kinetic models are used and compared.

Therefore, the objective of this study was to explore the synergistic effect and biodegradation kinetics of sewage sludge (SS) and food waste (FW) during the anaerobic co-digestion. Three classical kinetic models, i.e. modified Gompertz model [21], the two-substrate model [22] and cone model [23], were selected and used to analyze the experimental results in order to further understand biodegradation kinetics of the anaerobic co-digestion process. The theoretical methane yield in each SS:FW ratio was estimated based on the methane production of mono-digestion of SS or FW to evaluate the underlying synergistic effect. Besides, considering the environmental protection and treatment cost, the N_2O emissions and the dewaterability of digestate were discussed as well.

2. Material and methods

2.1. Substrates and inoculum

SS was collected from a wastewater treatment plant (WWTP) located in Shanghai, China. FW was sampled from a canteen (East China Normal University, Shanghai, China), most of which were rice, vegetables, and meat. The FW was diluted with tap water and homogenized by an electric blender (Yalixi, XP07, China) and was pre-screened through sieve of 10 mesh size to remove large particles. Then the collected samples were stored at 4 °C before use. The inoculum was obtained from an existing continuously stirred tank reactor (CSTR) fed with SS. The reactor has been running for roughly 100 days at 37 °C. Table 1 summarizes the main physicochemical properties of SS, FW and inoculum.

2.2. Biochemical methane potential (BMP) tests

The BMP tests were conducted in the 120-mL glass serum bottle with the working volume of 90 mL. The bottles were put in a constant temperature incubator shaker (37 ± 0.2 °C) with the vibration frequency of 130 rpm. Based on the dry VS, the seven groups of tests with different SS:FW ratios were carried out: 1.0:0.0 (i.e. mono-digestion of SS), 0.8:0.2, 0.6:0.4, 0.5:0.5, 0.4:0.6, 0.2:0.8 and 0.0:1.0 (i.e. mono-digestion of FW). The total VS of the substrate added in each bottle was 0.3 g. Then inoculum was added into each bottle and the ratio of inoculum to substrates was controlled at 2.0:.0 (based on VS). An equal amount of inoculum without the substrates was added to a serum vial to remove the background values of biogas production in each set of ratios. The tap water was added into each vial to reach the same working volume of 90 mL. Each test was done in parallel to ensure repeatability of the experiment. The pH of the co-substrate was neutralized to around 7.0 by buffer (5 mol-NaOH/L). The volume of buffer added in each sample was similar, ranging 0.1–1.0 mL. The bottles were then sealed with rubber stoppers and aluminum crimp and the headspace of bottles

Table 1
Characteristics of the substrates and inoculum.

Constituent	Sewage sludge (SS)	Food waste (FW)	Inoculum
pH	6.8 ± 0.0	4.4 ± 0.0	7.0 ± 0.0
TS (g/L)	45.3 ± 0.1	51.8 ± 0.4	30.2 ± 0.1
VS (g/L)	22.4 ± 0.1	47.1 ± 0.5	14.7 ± 0.0
TCOD (g/L)	25.6 ± 0.3	69.7 ± 3.8	25.8 ± 0.3
SCOD (g/L)	0.3 ± 0.0	45.5 ± 0.4	0.4 ± 0.0
Total protein (mg/L)	1393.1 ± 95.2	3129.5 ± 63.2	/
Soluble protein (mg/L)	41.7 ± 0.0	582.3 ± 31.6	/
Total carbohydrate (mg/L)	582.4 ± 86.4	13549.3 ± 129.9	/
Soluble carbohydrate (mg/L)	8.8 ± 0.8	11791.0 ± 1249.0	/
Ammonia nitrogen (mg/L)	143.4 ± 22.9	13.8 ± 0.4	/
Carbon/Nitrogen ratio (C/N)	6.3 ± 0.1	17.1 ± 0.0	/

Note: TS – Total solids; VS – Volatile solids; TCOD – Total chemical oxygen demand; SCOD – Soluble chemical oxygen demand.

was flushed with nitrogen gas for about 1 min in order to ensure the anaerobic condition. The experiment of co-anaerobic digestion continued until the biogas production was almost stagnant, which lasted for 58 days. During the process, the composition of the produced biogas (CH_4, CO_2 and N_2O) was collected by the glass syringe of 10–50 mL and measured periodically. The values of daily production and accumulative production of CH_4 and N_2O were calculated via the equation proposed by Zhen et al. [2] and then was adjusted to the standard conditions (273.15 K, 100.00 kPa). On day 0, 5, 17, 27 and 58, the supernatant of around 1 mL was taken by the plastic injector for further analysis.

2.3. Synergistic effect and kinetic simulation

Three mathematical models were used, including the modified Gompertz model (Eq. (1)) [21], two-substrate model (Eq. (2)) [22] and Cone model (Eq. (3)) [23] to study the digestive mechanisms of mixed wastes in anaerobic systems.

$$B_{(t)} = f_d \cdot \exp(-\exp[R_m e(\lambda - t)/f_d], \ t \geq 0) \quad (1)$$

$$B_{(t)} = B_{or} - B_{or}, e^{-krt} + B_{os} - B_{os} e^{-kst}, t \geq 0 \quad (2)$$

$$B_{(t)} = f_d/(1 + (k_{hyd}t)^{-n}), \ t > 0 \quad (3)$$

where B(t) – the specific methane yield at a given time (mL/g-VS); f_d – the maximum methane potential (mL/g-VS); λ – the lag-phase (day); R_m – the maximum methane production rate (mL/g-VS/day); t – the digestion time (day); e – the exponential e (2.71828); B_{or} – biochemical methane potential of the rapidly biodegradable substrates (mL/g-VS); B_{os} – biochemical methane potential of the slowly biodegradable substrates (mL/g-VS); k_r – hydrolysis rate of the rapidly biodegradable substrates (1/day); k_s – hydrolysis rate of the slowly biodegradable substrates (1/day); k_{hyd} – the hydrolysis rate constant (1/day); and n is the shape factor.

In order to investigate the synergistic effect of co-digestion on bioenergy recovery, the theoretical methane production in each ratio was estimated based on the methane production of mono-digestion of SS or FW (Eq. (4)) [24].

$$B_{estimated}(t) = B_{SS}(t) \times P_1 + B_{FW}(t) \times P_2 \quad (4)$$

where t – the digestion time (day); $B_{estimated}$ (t) – the estimated methane yield at the t day (mL/g-VS); B_{SS}(t) – the measured methane yield of SS alone at the t day (mL/g-VS); P_1 – the percentage of SS in the co-substrates (%); B_{FW} (t) – the measured methane yield of FW alone at the t day (mL/g-VS); and P_2 – the percentage of FW in the co-substrates (%).

2.4. Analysis methods

TS and VS were determined according to the Standard Methods [25]. The pH was measured with a pH meter (PHS-25, China). TCOD and SCOD were measured using the purchased HACH standard reagents according to the manufacturer's instructions. The protein was determined by the Folin-phenol reagent method (Lowry method) [26], the carbohydrate was measured by the phenol-sulfuric acid method [27], and the ammonia nitrogen was determined by the phenol-hypochlorite method [25]. Biogas composition (CH_4, CO_2 and N_2O) was analyzed by gas chromatography (Agilent 7890A, USA) equipped with a TCD detector. VFAs (volatile fatty acids) were measured using a gas chromatography (Agilent 7890GC-5975MS, USA) with a capillary column equipped with an FID. A capillary suction timer (Type 304M, England) with an inner diameter funnel (0.535 cm) and Triton CST paper (7 × 9 cm, Electronics Ltd, England) was used to measure the digestate dewaterability. The C/N ratio was measured by an elemental analyzer (vario MICRO cube, Germany) equipped with a TCD detector. Three-dimensional excitation–emission matrix (TD-EEM) spectroscopy of the digestate supernatant was determined by a F-7000 FL spectrophotometer (Hitachi, Ltd., Japan) at 12,000 nm/min (scanning speed). The scanning emission (E_m) wavelength varied from 200 to 400 nm with the varying excitation (E_x) wavelength from 250 to 550 nm. The Origin, Sigmaplot, Curve Expert 1.4, and Excel were used for data analysis and fitting.

3. Results and discussion

3.1. Solid organics degradation

The initial and final values of TS, VS, TCOD and C/N ratio under different SS:FW ratios were measured, and the degradation efficiency of the solid organics was calculated (Table 2). The initial VS was all controlled at 3.3 g/L, and the initial TS decreased with increasing proportion of FW due to higher VS/TS ratio in FW. The final TS and VS showed a downward trend along with the decreased proportion of SS. The removal rate of TS and VS, as given in Table 2, presented an upward trend with the decreased proportion of SS. It might be attributed to the complex sludge structure, such as rigid cell walls and extracellular biopolymers [2,24,28], which caused the poor degradation efficiency. The presence of refractory substances (light metal ions, heavy metals, etc.) in SS was also one of causes for such poor performance [29]. In contrast, FW, which is mainly composed of carbohydrates, proteins, lipids and vitamins, can be easily hydrolyzed and utilized. However, an increase in the FW content does not necessarily lead to a better removal efficiency. For example, the maximum TCOD removal rate of 76.6 ± 13.3% was obtained at the SS:FW ratio of 0.8:0.2, but it declined to 68.6 ± 6.3% at the SS:FW ratio of 0.0:1.0.

The C/N ratio directly affects the stability of the system [30]. It is reported that the C/N ratio less than 6.0 could produce toxicity to harm anaerobic microorganisms due to the low carbon availability and the high ammonia (NH_3) concentration. On the contrary, the C/N ratio higher than 30 could cause the low organic removal rate and the low methane production due to the nutrient deficiency [12,31]. The initial C/N ratio in mono-SS was 6.3 ± 0.1 while the initial value in mono-FW was around 17.1 ± 0.0. With the addition of FW, the initial C/N

Table 2
Variations of TS, VS, TCOD and C/N ratio under different SS:FW ratios during co-digestion.

VS_{SS}:VS_{FW}	1.0:0.0	0.8:0.2	0.6:0.4	0.5:0.5	0.4:0.6	0.2:0.8	0.0:1.0
$TCOD_{initial}$ (g/L)	3.8 ± 0.0	4.0 ± 0.0	4.2 ± 0.1	4.3 ± 0.1	4.4 ± 0.1	4.6 ± 0.2	4.8 ± 0.2
$TCOD_{final}$ (g/L)	1.8 ± 0.5	2.6 ± 0.3	1.6 ± 0.2	1.7 ± 0.0	1.6 ± 0.5	1.1 ± 0.6	1.5 ± 0.3
$TCOD_{RR}$ (%)	53.1 ± 14.1	35.2 ± 7.6	62.7 ± 5.5	59.8 ± 0.3	64.5 ± 12.1	76.6 ± 13.3	68.6 ± 6.3
$TS_{initial}$ (g/L)	6.8 ± 0.0	6.1 ± 0.0	5.5 ± 0.0	5.2 ± 0.0	4.9 ± 0.0	4.3 ± 0.0	3.7 ± 0.0
TS_{final} (g/L)	4.0 ± 0.1	2.7 ± 0.5	2.5 ± 0.1	2.0 ± 0.4	2.1 ± 0.2	1.4 ± 0.1	0.8 ± 0.2
TS_{RR} (%)	41.1 ± 1.8	56.7 ± 7.6	55.2 ± 0.9	62.0 ± 6.8	57.8 ± 5.0	66.5 ± 2.5	77.3 ± 4.8
$VS_{initial}$ (g/L)	3.3 ± 0.0	3.3 ± 0.0	3.3 ± 0.0	3.3 ± 0.0	3.3 ± 0.0	3.3 ± 0.0	3.3 ± 0.0
VS_{final} (g/L)	1.2 ± 0.0	1.0 ± 0.3	1.1 ± 0.1	1.1 ± 0.1	1.1 ± 0.2	0.6 ± 0.1	0.5 ± 0.0
VS_{RR} (%)	43.0 ± 0.8	69.1 ± 10.3	67.2 ± 2.2	68.2 ± 2.0	68.2 ± 4.5	81.5 ± 2.4	85.2 ± 0.4
$C/N_{initial}$	6.3 ± 0.1	7.5 ± 0.1	8.9 ± 0.0	9.8 ± 0.0	10.8 ± 0.0	13.4 ± 0.0	17.1 ± 0.0
C/N_{final}	6.0	6.1	6.4	6.3	6.5	6.5	6.5

Note: RR – removal rate.

ratio showed a certain degree increase, similar to the findings obtained by Liu et al. [32]. It's worth noting that the values of C/N ratio in all scenarios were below the optimal range (20–30) of anaerobic digestion. The addition of FW increased C/N ratio and provoked the final methane yield, but too high dosage gave rise to slower hydrolysis rate and longer lag-phase. Amongst all the studied SS:FW ratios, the 0.5:0.5 ratio, which resulted in a C/N ratio of 9.8 ± 0.0, had the fastest hydrolysis rate and the shortest lag-phase.

3.2. Soluble organics liberation and re-consumption

The soluble organics (SCOD, soluble protein and soluble carbohydrate) were measured throughout the experiment, and the results are illustrated in Fig. 1a–c. The solubilization of the solid organics is considered as the rate-limiting step of the hydrolysis processes [33]. In the early stage of co-digestion (0–5 day), the SCOD concentration decreased at the SS:FW ratios of 1.0:0.0, 0.8:0.2, 0.5:0.5, and 0.4:0.6, but the significant increase was observed at the SS:FW ratios of 0.2:0.8 and 0.0:1.0 (Fig. 1a). It showed that the excessive increase in the proportion of FW could lead to the decrease of soluble organics utilization rate, thereby reducing the digestion efficiency. The phenomenon was consistent with the variations of methane production. Subsequently, the SCOD concentration began to increase for all SS:FW ratios (6–17 day) and then sharply deceased to nearly 0 mg/L (18–58 day). This stage might involve multiple complex processes such as cell lysis, solid dissolution, and conversion and utilization of soluble organics by microorganisms. Besides, the soluble carbohydrate showed the similar tendency with SCOD while the variation of soluble protein was different, and the protein concentration had been declining, approaching 0 mg/L on day 20 (Fig. 1b and c). The difference might be caused by different binding forces. Many researches have demonstrated that carbohydrates had the strong binding force with lignin (e.g. cellulose and hemicellulose) [34,35], as a result of which carbohydrates had more difficulty to hydrolyze.

The pH and VFAs are the important parameters reflecting the stability of the system and the variation of pH and VFAs could affect the activity of methanogenic bacteria [36]. Fig. 1d illustrates the variation of pH under different SS:FW ratios. The initial and final pH were in the range from 6.8 to 7.2, which is suitable range for the growth of anaerobic microorganisms [37]. It showed that the system was relatively stable throughout the whole process of co-digestion. Moreover, as shown in Fig. 1e, the lowest VFAs concentration (26.55 mg/L) was observed at the SS:FW ratio of 0.5:0.5 on day 5. With the increased proportion of FW, the VFAs concentration increased dramatically, and the highest value of 1577.2 mg/L was observed at the SS:FW ratio of 0:1.0. On day 27, the VFAs concentration of the mono-FW declined to 799.64 mg/L. Correspondingly, the methane production of the 0.0:1.0 ratios had improved during this period. It showed that the VFAs accumulation had a certain degree impact on anaerobic system stability. Similarly, Duan et al. [38] reported that the 1000–3000 mg-VFAs/L could cause the moderate inhibition. Hence, excessive FW was not conducive to digestion performance, and it could reduce the hydrolysis rate and prolong the lag-phase to a certain degree. In addition, the decrease of VFAs concentration and the increase of final pH demonstrated the relationship that the accumulation of VFAs could cause the decrease of pH, which was consistent with some researchers' statement [28,39].

3.3. Methane production

Methane production rate and accumulative methane yield for co-digestion of SS and FW under different SS:FW ratios are displayed in

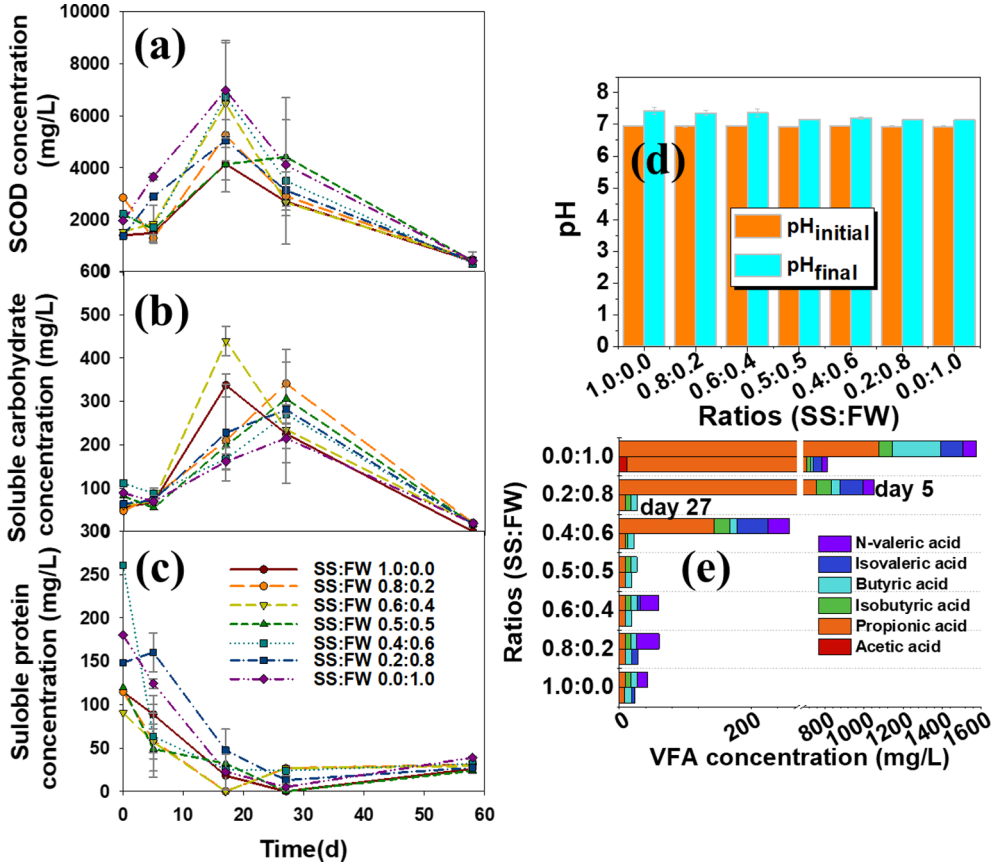


Fig. 1. Variations of soluble organics, pH and VFA during co-digestion under different SS:FW ratios after 58 day of co-digestion: (a) SCOD, (b) soluble carbohydrate, (c) soluble protein, (d) pH and (e) VFAs.

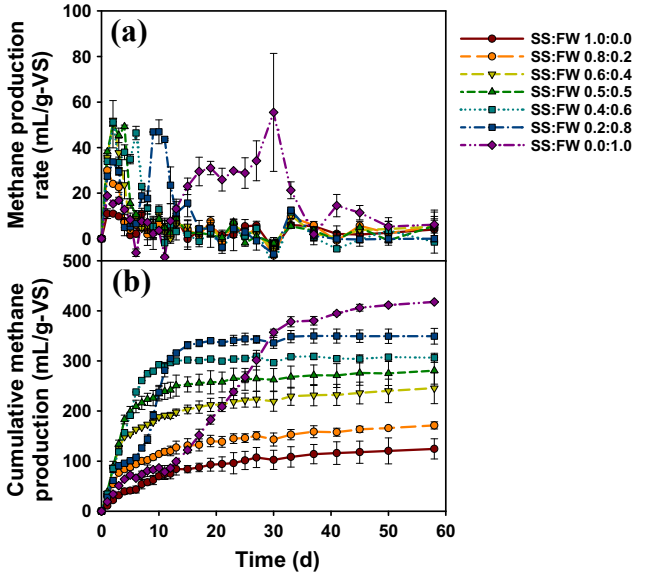


Fig. 2. Methane production rate (a) and accumulative methane yield (b) over time during co-digestion under different SS:FW ratios.

Fig. 2. For the mono-digestion of SS, the highest methane production rate was only 10.97 ± 2.12 mL/g-VS/day. In contrast, co-digestion with FW increased the methane production. For example, the highest methane production rate was 29.96 ± 0.95 mL/g-VS/day at 0.8:0.2 ratio, which was 2.73-fold than that of alone SS; and it increased to 52.01 ± 0.67 mL/g-VS/day at 0.6:0.4 ratio; then to 50.30 ± 10.37 mL/g-VS/day at 0.5:0.5 ratio. It was worth noting that the methane production rate had not been further improved when the FW proportion was higher than 40%. Conversely, the excessive addition might lead to the prolonged lag-phase. For the 0.5:0.5 ratio, the highest methane production rate was achieved at day 2; for the 0.2:0.8 ratio, it appeared on day 10 (46.93 ± 5.23 mL/g-VS/day) while for the FW alone, it was attained on day 30 (55.46 ± 25.92 mL/g-VS/day). This might be due to the high content of lipid and some refractory substances present in FW, which required longer hydrolysis and digestion time [12,40].

Fig. 2b shows that the accumulative methane yield of the mono-digestion of SS was lowest, only 124.43 ± 20.10 mL/g-VS. The similar results for SS mono-digestion were also reported by Kim et al. [41] (116 mL/g-VS_{deg}) and Zhen et al. [2] (176.36 ± 0.00 mL/g-VS_{added}). By contrast, the final methane yield increased with the proportion of FW, and the highest yield of 417.72 ± 0.94 mL/g-VS was observed at sole FW digestion, which was 3.36-fold than that of mono-SS. The studies of Liu et al. [32] and Heo et al. [42] on anaerobic co-digestion of SS and FW also reported that the biogas production increased with increasing proportion of FW. However, the accumulative methane yield

Table 3
Estimated parameters of modified Gompertz, two-substrate and Cone models.

Model	Parameters	VS_{SS}:VS_{FW}						
		1.0:0.0	0.8:0.2	0.6:0.4	0.5:0.5	0.4:0.6	0.2:0.8	0.0:1.0
	$B_{measured}$ (mL/g-VS)	124.42	171.17	245.53	280.60	306.24	349.22	417.72
Modified Gompertz model	H_m (mL/g-VS)	115.54	155.79	221.75	260.81	305.12	354.18	465.15
	R_m (mL/g-VS/day)	5.50	9.50	23.13	39.47	46.50	28.71	12.26
	λ (/day)	2.098	3.288	1.370	0.182	0.456	1.382	3.182
	$B_{predicted}$ (mL/g-VS)	115.40	155.77	221.75	260.81	305.12	354.18	440.93
	R^2	0.9778	0.9734	0.9752	0.9868	0.9943	0.9816	0.9836
	Adjusted R^2	0.9760	0.9706	0.9726	0.9854	0.9937	0.9797	0.9822
	Diff. (%)	7.23	9.01	9.67	7.05	0.35	1.42	5.57
	S.E.E.	5.49	8.54	12.56	11.18	8.73	18.18	18.80
	RSS (1 0 ^{3})	0.7229	2.6160	6.0573	3.8492	0.6491	10.5022	8.4805
	AIC	98.58	133.30	155.97	143.73	95.67	170.83	165.06
	rMSPE	4.33	7.38	13.21	11.07	4.38	19.77	17.44
Two-substrate model	f_d (mL/g-VS)	149.01	171.01	249.70	323.05	310.52	366.99	793.20
	B_r (mL/g-VS)	81.89	70.26	163.03	256.98	180.99	214.47	407.48
	B_s (mL/g-VS)	65.92	101.92	93.71	78.10	154.74	182.34	394.78
	K_r(1/day)	0.1172	0.5208	0.3945	0.2856	0.2290	0.1083	0.0156
	K_s(1/day)	0.0171	0.0579	0.0458	0.0104	0.2290	0.1083	0.0156
	$B_{predicted}$ (mL/g-VS)	124.42	167.46	243.11	280.43	310.52	366.25	468.92
	R^2	0.9959	0.9949	0.9904	0.9986	0.9847	0.9595	0.9603
	Adjusted R^2	0.9951	0.9939	0.9885	0.9863	0.9816	0.9512	0.9531
	Diff. (%)	0.01	2.18	0.97	0.06	1.41	4.88	12.28
	S.E.E.	2.47	3.91	8.15	10.82	14.92	28.17	30.52
	RSS (1 0 ^{3})	0.1344	0.1671	0.5510	1.1035	3.5386	22.6263	20.4957
	AIC	59.54	65.42	97.63	116.38	147.84	197.94	195.27
	rMSPE	2.31	2.50	3.82	5.45	10.25	28.65	27.50
Cone model	f_d (mL/g-VS)	147.50	201.14	249.27	274.69	311.63	363.70	597.99
	k_{hyd} (1/day)	0.086	0.143	0.289	0.334	0.277	0.131	0.034
	n	1.035	0.778	1.064	1.564	2.195	2.447	1.643
	$B_{predicted}$ (mL/g-VS)	124.01	168.61	237.45	272.05	310.93	361.18	452.42
	R^2	0.9957	0.9957	0.9909	0.9951	0.9917	0.9582	0.9697
	Adjusted R^2	0.9953	0.9953	0.9901	0.9947	0.9910	0.9548	0.9671
	Diff. (%)	0.32	1.51	3.28	3.05	1.54	3.43	8.32
	S.E.E.	2.41	2.89	6.00	5.29	8.64	26.06	25.56
	RSS (1 0 ^{3})	0.1403	0.2003	0.8650	0.6723	1.7900	16.2989	15.6827
	AIC	54.31	63.93	103.42	96.62	123.06	182.70	181.66
	rMSPE	2.38	2.63	5.21	4.82	8.40	24.65	24.05

Note: Diff. (%) = $|B_{measured} - B_{predicted}|/B_{measured} \times 100$). S.E.E. (standard error of estimate), RSS (residual sum of squares), rMSPE (root mean square prediction error) and AIC (akaike's information criterion) are calculated based on the method proposed by Zhen et al. [6].

in all ratios varied in different period due to the difference in substrate compositions. For example, the 0.5:0.5 ratio achieved the accumulative methane yield of 198.51 ± 10.68 mL/g-VS on day 5, increasing by 4.85-, 2.75-fold than that of single SS and FW, respectively. It demonstrated the important role played by co-digestion in promoting the degradation of biowastes and bioenergy recovery.

3.4. Kinetic modeling of co-digestion process

Many factors, such as types and properties of substrates, experimental conditions, operating parameters, and structure of reactor, could influence the accuracy and reliability of the model [2]. Multiple models could be beneficial to ensure the authenticity of the fitting data. Hence, three models of modified Gompertz model, two-substrate model and Cone model were selected and used to fit the methane production achieved from BMP tests in order to explore the degradation kinetics of co-substrate. Table 3 summarizes the kinetic parameters of three models. According to the analysis of the three models, the predicted maximum methane potential all showed an increase with the increased proportion of FW in feedstock. It was in line with the variation of actual methane production. However, it does not mean that the higher the proportion of FW, the better the performance of the co-digestion system.

The maximum methane production rate (R_m) and the lag-phase (λ) were estimated by the modified Gompertz model. The R_m of 0.5:0.5 ratio was 39.47 mL/g-VS/day, which increased by 7.12- and 3.22-fold compared with the SS and FW mono-digestion, respectively. Although it was slightly lower than that of 0.4:0.6 ratio (46.50 mL/g-VS/day), the λ of 0.5:0.5 ratio (0.182 day) was lowest, which was shortened by 11.53-, 2.51 - and 17.50-fold than that of 1.0:0.0 ratio, 0.4:0.6 ratio and 0.0:1.0 ratio, respectively. In addition, hydrolysis was considered to be the rate limiting stage of anaerobic digestion process [6,43], which could influence degradation efficiency and methane production. Hence, the hydrolysis rate was predicted by the two-substrate model and Cone model. The result of Cone model showed that the 0.5:0.5 ratio had the highest hydrolysis rate (0.334/day), which was 3.88- and 9.82-fold than that of 1.0:0.0 ratio (0.086/day) and 0.0:1.0 ratio (0.034/day) respectively. However, the result of two-substrate model indicated the 0.2:0.8 possessed the highest rapid hydrolysis rate (0.52/day). The difference might be contributed to the accuracy and reliability of different models. Interestingly, the two model all demonstrated the hydrolysis rate of co-digestion was higher than that of mono-digestion. The results reflected the co-digestion system could create a suitable metabolism circumstance and it was beneficial to the activity of methanogens [24], thereby improving the biodegradability and conversion rate of co-substrate.

In order to further ascertain the reliability of three models, the Diff, R^2, adjusted R^2, S.E.E., RSS, rMSPE and AIC were calculated (Table 3), and the Pearson's correlation between the measured final methane yield ($B_{measured}$) and the predicted final methane yield ($B_{predicted}$) of three models is displayed in Fig. 3a. The value of Diff measured by Cone model was lowest (0.32–8.32%), followed by modified Gompertz model (0.35–9.67%) and two-substrate model (0.01–12.28%). According to the calculated values of R^2, adjusted R^2, S.E.E., RSS, rMSPE and AIC, it was obvious that the two-substrate model had the lowest suitability and precision. The result demonstrated that the Cone model was more reliable for the above-mentioned analysis of hydrolysis rate. Moreover, the Pearson's correlation of Cone model was relatively high (0.9957). On the whole, the Cone model best fitted the actual evolution of methane production. Some researchers [2,6,44] also confirmed the Cone model had the highest suitability and precision.

Subsequently, the quadric correlation analysis between FW percentage (χ, %) and hydrolysis rate constant (k_{hyd}), λ were performed in order to ascertain the role of FW in biodegradation kinetics and methane production of SS. Fig. 3b and c shows that the FW percentage (χ, %) with k_{hyd}, and λ had high quadric correlations (b: $k_{hyd}(\chi) = 0.08 + 0.26\chi + 0.53\chi^2$, $R^2 = 0.9854$; and $\lambda(\chi) = 2.15 + 10.71\chi - 29.91\chi^2$, $R^2 = 0.9723$; c: $k_{hyd}(\chi) = 0.79 - 1.04\chi + 0.28\chi^2$, $R^2 = 0.9966$; and $\lambda(\chi) = 2.04 - 8.46\chi + 9.60\chi^2$, $R^2 = 0.9994$; respectively). With the increase of FW percentage in SS from 0 to 50%, the k_{hyd} increased, and from 50 to 100%, the k_{hyd} began to decrease. In general, the increase of k_{hyd} could cause the decrease of λ, However, for FW percentage in SS from 0 to 20%, the k_{hyd} increased while the λ also increased. It indicated that some factors except for the hydrolysis rate also could influence the degradation efficiency of co-digestion. The works to explore the role of other factors in affecting degradation efficiency are needed in future. Overall, the 50% FW percentage in SS (i.e. 0.5:0.5) had the better performance efficiency, which possessed the highest k_{hyd} and the lowest λ, It further confirmed the high feasibility of co-digesting SS and FW, and the current work will provide an important reference for the practical applications of co-digestion technology in biomass co-treatment and energy recovery.

3.5. Co-digestion synergistic impact

Based on the Eq. (4), the methane production in all the scenarios was estimated. By comparing the measured methane production with

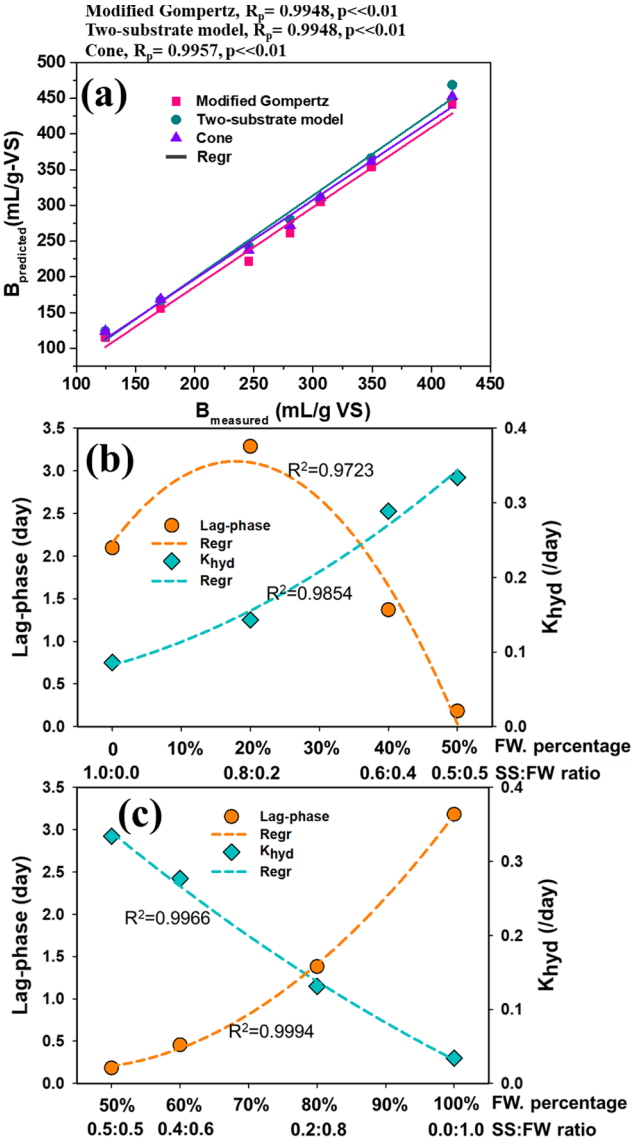


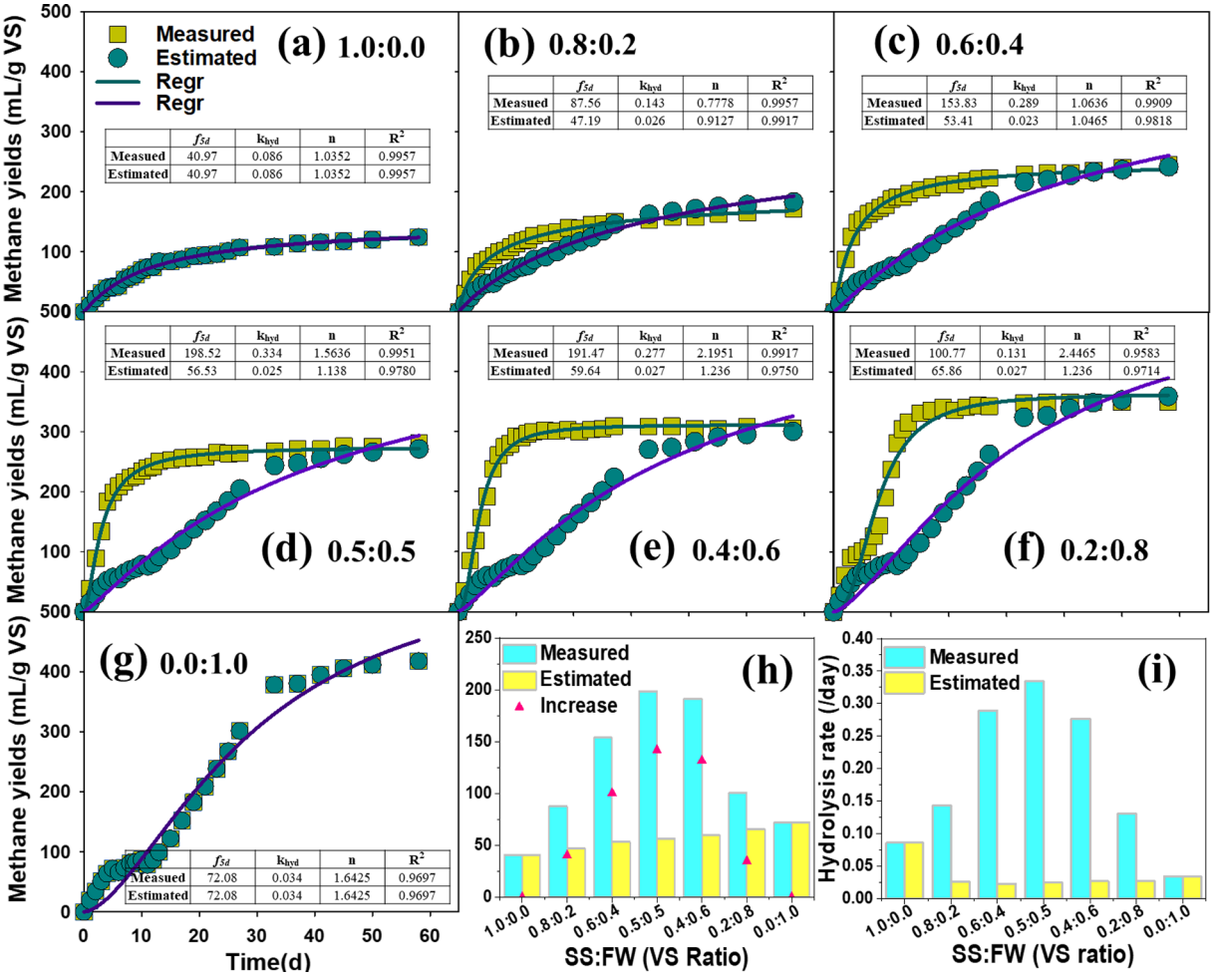
Fig. 3. Pearson's correlations between the measured final methane yield ($B_{measured}$) and the predicted final methane yield ($B_{predicted}$) by three classical model (a), and quadric correlations between k_{hyd}, λ and FW percentage (or SS:FW ratio): FW percentage 0–50% (b), FW percentage 50–100% (c).

the estimated methane production, the synergistic impact of co-digested SS and FW was studied (Fig. 4a–g). The difference in methane production rate of the actual and the theoretical level proved the existence of the synergistic impact and its positive role during the co-digestion. For example, on day 5, the 0.5:0.5 ratio showed the highest increase of 142.00 mL/g-VS, which was up to 198.52 mL/g-VS (Fig. 4h). Then the synergistic effect attenuated with the increasing FW, and the corresponding value decreased to 131.83 mL/g-VS at 0.4:0.6 ratio and then to 34.91 mL/g-VS. The positive synergistic impact might be attributed to the enhancement of buffering ability and the improvement of anaerobic microorganisms' activity due to the supplement of macro or micro nutrients [12,24,44]. By comparison, the 0.5:0.5 could create a more suitable anaerobic environment, which was beneficial to the methane conversion. It indicated that the synergistic effect was not positively related to the increasing proportion of FW. Similar to the discovery of Zhen et al. [6], they reported that the MA (microalgae):FW (food waste) ratio of 0.5:0.5 had the highest increase of roughly 54%, but not the expected ratio of 0.2:0.8.

Meantime, the Cone model was used to fit the actual and theoretical methane data in order to further explore the kinetic mechanism caused by synergistic effect, and the corresponding parameters are summarized in Fig. 4. The k_{hyd} of the actual methane production was much higher than the k_{hyd} of the theoretical methane production (Fig. 4i). The gap at 0.5:0.5 ratio was biggest, and the k_{hyd} of the actual data was 13.36-fold compared with that of the theoretical data. It suggested that the hydrolysis played an important role in the degradation efficiency. The synergistic effect involved in co-digestion process improved hydrolysis efficiency, thereby upgrading the degradation efficiency and methane production, which was in agreement with the fact elaborated in Section 3.4. In addition, Koch et al. [45] co-digested raw sludge and FW, and they also found that the co-digestion improved the hydrolysis rate, thereby accelerating the degradation and methane production. Thus, co-digestion can be a useful way to improve hydrolysis efficiency for enhancing the performance of anaerobic system.

3.6. Ammonia nitrogen release and N_2O emissions

Ammonia nitrogen has an impact on the stability of the anaerobic system. The high ammonia nitrogen concentration could cause the high concentration of free ammonia, which could inhibit the activity of methanogenic enzyme [29,46]. Hence, the variations of ammonia nitrogen concentration during the process of anaerobic co-digestion was detected (Fig. S2a in Supplementary Information). The result shows that the ammonia nitrogen concentration increased overtime for all SS:FW ratios. The rise in ammonia nitrogen concentration might be ascribed to the degradation of proteins or other nitrogen-containing organic substances [47]. The highest ammonia nitrogen concentration was obtained for the SS mono-digestion at the end of the experiment, up to 400.15 ± 0.85 mg/L. The corresponding value of other ratios was all around 380 mg/L. It confirmed that the low C/N ratio of SS could produce the high ammonia (NH_3). FW with high C/N ratio added in SS could reduce the accumulation of ammonia nitrogen. Moreover, due to the difference in types of substrates and operation conditions, the inhibition threshold of ammonia concentration might be different in



	f_{5d}	k_{hyd}	n	R^2
Measured	40.97	0.086	1.0352	0.9957
Estimated	40.97	0.086	1.0352	0.9957

	f_{5d}	k_{hyd}	n	R^2
Measured	87.56	0.143	0.7778	0.9957
Estimated	47.19	0.026	0.9127	0.9917

	f_{5d}	k_{hyd}	n	R^2
Measured	153.83	0.289	1.0636	0.9909
Estimated	53.41	0.023	1.0465	0.9818

	f_{5d}	k_{hyd}	n	R^2
Measured	198.52	0.334	1.5636	0.9951
Estimated	56.53	0.025	1.138	0.9780

	f_{5d}	k_{hyd}	n	R^2
Measured	191.47	0.277	2.1951	0.9917
Estimated	59.64	0.027	1.236	0.9750

	f_{5d}	k_{hyd}	n	R^2
Measured	100.77	0.131	2.4465	0.9583
Estimated	65.86	0.027	1.236	0.9714

	f_{5d}	k_{hyd}	n	R^2
Measured	72.08	0.034	1.6425	0.9697
Estimated	72.08	0.034	1.6425	0.9697

Fig. 4. Measured and estimated methane production (a–g), and the increase in measured methane yields over estimated at 5 day (h) as well as the k_{hyd} variation of measured and estimated methane dates (i) during co-digestion under different SS:FW ratios.

different researches. Benabdallah El Hadj et al. [48] found that 215 mg-NH_3-N/L inhibited the biomethane production under mesophilic conditions. Angelidaki and Ahring [49] reported the inhibition threshold of 700 mg/L. In this study, the decline of methane production indicated that high ammonia nitrogen (over 300 mg/L) might slightly inhibit the methanogenic activity.

Besides, N_2O is one of the intermediates during the denitrification process [50,51], and its ozone-depleting potential is 298 times than that of the equivalent mass of CO_2 [52]. Accordingly, the N_2O emissions during the co-digestion process was measured (Fig. S2b and c in Supplementary Information). The N_2O emissions for SS mono-digestion was the lowest, which was $5.32 \times 10^{-5} \pm 2.45 \times 10^{-7}$ mL/g-VS. With the increased proportion of FW, the N_2O emissions had a slight increase. However, for the alone FW digestion, the N_2O emissions had a dramatic increase ($2.15 \times 10^{-4} \pm 9.42 \times 10^{-6}$ mL/g-VS), which was 4.04-fold than that of mono-digestion of SS. The high N_2O emissions might be ascribed to the metabolism imbalance of the microorganisms responsible for nitrification or denitrification. The addition of SS into FW improved the metabolic environment, thus reducing the N_2O emissions. He et al. [53] also reported the high N_2O emissions potential of FW during the digestion. The results showed the alone FW digestion had much greater potential for N_2O emissions.

3.7. Three-dimensional fluorescence spectroscopy for digestate supernatant

TD-EEM spectroscopy of the dissolved organic matter (DOM) fractions in digestate supernatant collected from different SS:FW ratios was measured, and the parallel factor (PARAFAC) analysis was used to explore the fluorescence properties. The results are illustrated in Fig. 5, and two components were detected in digestate supernatant. The excitation/emission wavelength (E_x/E_m) of component 1 were centered at 225/355 nm (peak A) and 280/355 nm (peak B), and component 2 was located at the E_x/E_m of 250/450 nm (peak C) and 330/450 nm (peak D) (Fig. 5a and b). According to Coble [54] and Kwon et al. [55], the peak A was ascribed to the aromatic protein-like substances, the peak B was caused by tryptophan protein-like substances, and the peak C and peak D were assigned to continental humic-like substances. Compared with the similar peaks in the effluent samples extracted from a leachate-fed EGSB bioreactor reported by Lu et al. [56], the locations of the peaks occurred a slight shift. Main reason might be the interactions between protein-like and humic-like components or other undetected components [57].

In general, the protein-like substances are derived from free amino acids, carbohydrates, proteins or peptides [55]. The cell lysis also produces the protein-like substances [56]. The humic-like substances

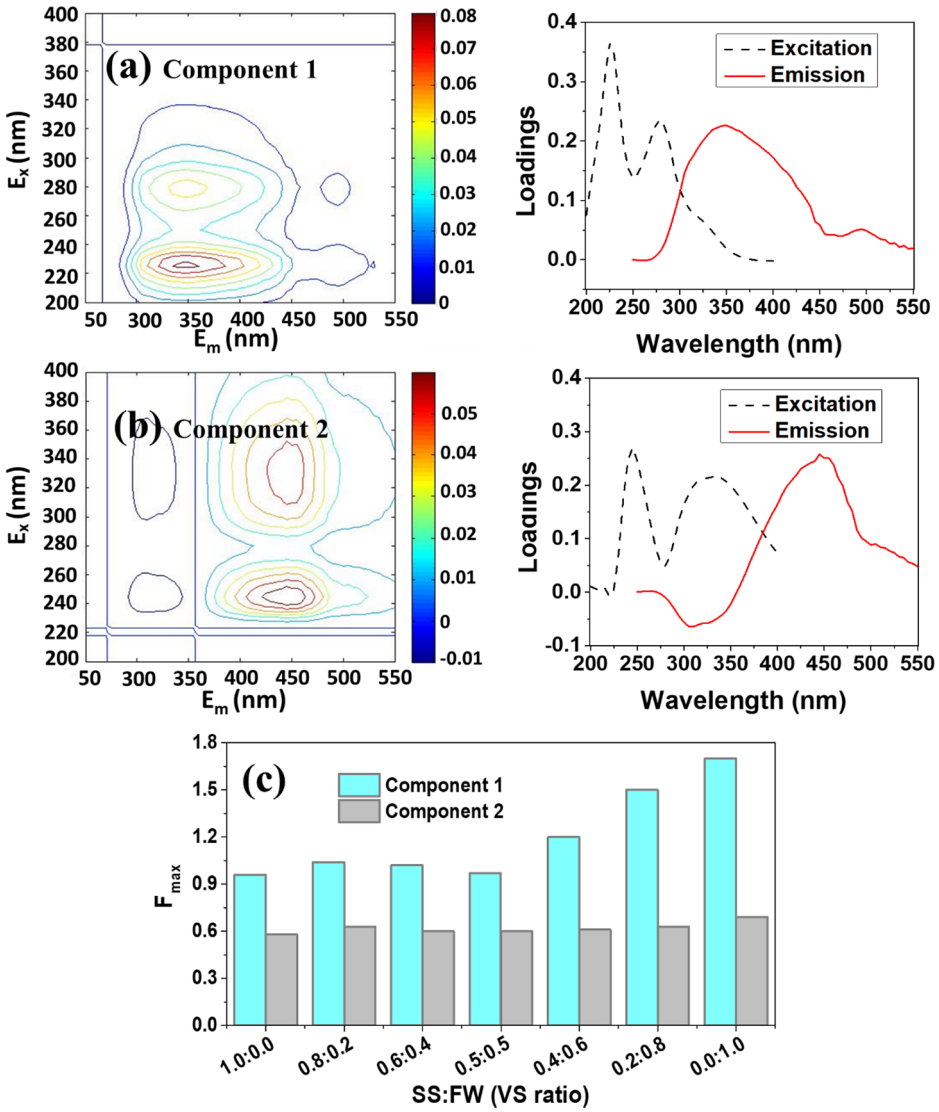


Fig. 5. TD-EEM contour plots of identified using the DOMFluor-ARAFAC model and the split-half validation model: component 1 (a), component 2 (b), and the maximum fluorescence intensities (F_{max}) in the two components collected from the digestate samples under different SS: FW ratios at the end of the experiment (c).

are produced by the biodegradation of organic matter, and the process of continental humic-like substances generation is more complex. These substances have a close relationship with the overall performance of anaerobic system. Fig. 5c displays the value of the maximum fluorescence intensities (F_{max}) of each component under different ratios. Overall, the F_{max} value of the two components first dropped and then rose with the increasing proportion of FW. It dropped the minimum at 0.5:0.5 ratio (1.0 and 0.6, respectively), explaining the rapid hydrolysis and short lag-phase observed at this ratio.

3.8. Digestate dewaterability

Digestate dewaterability in subsequent processing is of great significant parameter, which is related to the reduction of digestate, and the cost of treatment and disposal [58]. In order to explore the variation of digestate dewaterability in all scenarios, the capillary suction time (CST) was measured at the end of experiment (Fig. S3 in Supplementary Information). Overall, the CST of digestate with the addition of FW was much lower than that of SS mono-digestion. It demonstrated the addition of FW into SS improved the dewaterability of the digestate. The main reason might be the high content of the easily biodegradable substances (protein, carbohydrate, etc.) in FW. Hence, the addition of FW was also beneficial to digestate dewatering, reduction and final disposal.

4. Conclusions

The experimental results confirmed the superiority of anaerobic co-digestion of sewage sludge and food waste, and the suitable addition of food waste could improve the stability of system and upgrade the methane production. The SS:FW ratios of 0.5:0.5 achieved the high methane recovery, with the minimum lag-phase (0.182 day) and the maximum hydrolysis rate (0.334/day). Correlation model parameter analysis showed the Cone model had the best fitness to the experimental results. The improvement of hydrolysis efficiency induced by the synergistic effects was one of the most important reasons that boosted the methane production. Besides, the co-digestion of sludge with food waste was able to mediate the N_2O emissions while simultaneously improving the digestate dewaterability.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.fuel.2019.04.084.

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