Influence of Moisture Content of Solid-state NaOH Pretreatment and Codigestion on Methane Production in the Semi-dry Anaerobic Digestion of Rose Stalk

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Large quantities of burned or abandoned rose stalks are leading to serious environmental pollution. In this study, the effect of the moisture content of a solid-state NaOH pretreatment on methane production was first determined by a biochemical methane potential test. Then, the effect of codigestion with pig manure on methane production was investigated under the optimal moisture *via* thermophilic semi-dry anaerobic digestion by leaching bed reactor. Biogas production kinetic was assessed by the first-order kinetic model and modified Gompertz model. An increase in methane yield and biogas production kinetics was shown in the solid-state NaOH pretreated biomass. There was no significant difference in methane production for the three moisture contents studied during pretreatment (54%, 70%, and 77%). The anaerobic codigestion of rose stalk and pig manure increased 41% to 52% for methane yields and improved biogas production kinetics compared with monodigestion of rose stalk. Anaerobic codigestion did not greatly change the process stability, except for NH_4^+-N. The optimal process for the anaerobic digestion of rose stalk was as follows. The rose stalk was initially pretreated *via* solid-state NaOH pretreatment with a moisture content of 70%. Then, the pretreated rose stalk was co-digested with pig manure at a total solids ratio of 1:1.

Keywords: Moisture content; Solid-state NaOH pretreatment; Codigestion; Semi-dry anaerobic digestion; Kinetic assessment

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INTRODUCTION

The Kunming City of the Yunnan Province is the largest production base of rose (*Rosa rugosa*) in China. Every year in the region large quantities of rose stalks are either burned or abandoned in the field, thereby resulting in serious environmental pollution in Dianchi Lake (Liang *et al.* 2016). Therefore, the effective utilization of rose stalks could help restore the environmental quality of Dianchi Lake. The anaerobic digestion of organic compounds for bioenergy production is the most cost-effective and one of the cleanest energy options (Liotta *et al.* 2014; Mirmohamadsadeghi and Karimi 2018). Liang *et al.* (2016) recently reported that the methane yield of rose stalk reached 78.1 to 112.7 mL/g total solids (TS), equal to 18.4% to 26.5% of the theoretical maximum yield of methane (425.2 mL/g TS) for the semi-dry anaerobic process after 30 days of anaerobic digestion. This low methane production efficiency limits the economic feasibility of anaerobic digestion. Thus, the operational parameters of anaerobic digestion need to be optimized to improve process performance.

The lignocellulosic characteristics of rose stalk are resistant to the degradation rate and methane production (Liang *et al.* 2016). NaOH pretreatment is a promising technology for improving the biodegradability of lignocellulosic biomass (Hendriks and Zeeman 2009). Pang *et al.* (2008) proposed a solid-state NaOH pretreatment for lignocellulosic biomass and found that NaOH pretreatment with 6% (w/w) loading produced 48.5% more biogas yield. Thus, Liang *et al.* (2016) reported that 4% NaOH-treated rose stalk produced 44% higher yields than non-NaOH-treated biomass. The moisture content of pretreatment has a remarkable influence on pretreatment performance (Yuan *et al.* 2014; Peces *et al.* 2015). Moreover, the disruption of lignocellulosic structure and removal of chemical composition were affected by the moisture content during pretreatment, thereby changing the biogas production and process stability for anaerobic digestion of pretreated biomass (Yuan *et al.* 2014). However, no systematic study on the influence of moisture content on the performance of solid-state pretreatment has been conducted yet.

In general, wet anaerobic digestion has less than 10% TS in an anaerobic reactor, compared to about 10 to 15% TS for semi-dry anaerobic digestion, and greater than 15% TS for dry anaerobic digestion (Liang *et al.* 2016). The anaerobic codigestion of lignocellulosic biomass and organic wastes that contain high nitrogen has also been considered to be an efficient measure for improving methane production efficiency (Mehryar *et al.* 2017; Awais *et al.* 2018). For example, Marchetti *et al.* (2016) found that the anaerobic codigestion of pig slurry and wetland biomass improved the methane production potential of wetland biomass. Moreover, Tsapekos *et al.* (2017) recently reported that the anaerobic codigestion of ensiled meadow grass and manure changed the process performance and microbial community. However, information on co-digesting rose stalk with pig manure is limited.

The objective of this study is to first determine the effect of solid-state pretreatment moisture content on methane production, and then investigate the effect of codigestion with pig manure on the process performance of under optimal pretreated moisture. Methane production, kinetic assessment, and changes in pH, volatile fatty acids (VFAs), chemical oxygen demand (COD), and total alkalinity (TA) were determined.

EXPERIMENTAL

Materials

Rose stalk and inoculums

The rose stalks were collected from Jinning County in Kunming City, China and then chopped into 3 to 4 cm lengths using shears after air-drying.

Table 1. Chemical Characteristics of the Feedstock and Anaerobic Culture

Sample	Anaerobic Culture	Rose Stalk	Pig Manure
TS (%)	4.37	91.41	29.62
VS (%TS)	43.5	92.91	89.09
pH	7.82	/	9.00
C (%)	/	46.23	38.82
N (%)	/	1.57	2.73
H (%)	/	5.90	5.32
O[a] (%)	/	46.3	53.13
C/N	/	29.4	14.2
[a] Calculated by difference, VS- volatile solids			

The anaerobic culture was obtained from a laboratory-scale anaerobic reactor and subsequently concentrated before its use as inoculum. The pig manure was obtained from a large-scale pig farm in the Anhui Province, Hefei, China and then stored in the dark at 4 °C. Table 1 shows the chemical characteristics of the feedstock and anaerobic culture.

Experimental Design

The first experiment was conducted in batch reactors (1-L jar) to obtain the optimal moisture content of pretreatment. Firstly, 50 g of dried rose stalk was initially pretreated using 4% NaOH (w/w) at 55 °C for 3 days. During pretreatment, three ratios of dried rose stalk and distilled water (1:1, 1:2, and 1:3; w/w) were assessed, and their corresponding moisture content was 54%, 70%, and 77%, respectively. Secondly, biochemical methane potential (BMP) was assessed at 55 °C for four kinds groups, including the untreated biomass (CK group) and three pretreated biomasses with different pretreated moisture content by using a similar Angelidaki protocol (Angelidaki *et al.* 2009). The other three experimental groups that corresponded to the different moisture contents of NaOH pretreatment were referred to as R1 (54% moisture content), R2 (70% moisture content), and R3 (77% moisture content). The TS concentrations of the reactor for the four groups were maintained at approximately 12% by supplementing distilled water. Each experiment was conducted in triplicate.

The second experiment for the anaerobic codigestion experiment was conducted in a leaching bed reactor (LBR) with an inner diameter, height, and working volume of 50 cm, 13 cm, and 4.0 L, respectively. The structure of the leaching bed reactor was similar to that of a previous study (Liang *et al.* 2011). The four TS ratios of rose stalk and pig manure (w/w) were 1:0 (CK), 1:1 (A1), 1:2 (A2), and 1:3 (A3). The ratios were used to determine the effect of anaerobic codigestion on methane production. The rose stalk was pretreated at 4% NaOH (w/w) with a rose stalk and distilled water ratio of 1:2 at 55 °C for 3 days before anaerobic digestion. The initial weight of the total solids on all digested biomass was 130 g for all reactors. The total solid reactor concentrations for the four groups were maintained at approximately 11.5% by supplementing distilled water. All LBRs were placed into an incubator at 55 ± 1 °C for anaerobic digestion. The 400-mL capacity of the leaching liquid was recycled to the top to sprinkle the bed twice a day. A 5 mL-sample was first taken out from leaching liquid for analysis before recirculation, and then distilled water was supplied leachate so as to reach 400 mL (Liang *et al.* 2011). The leachate sample was used as the analysis of COD, NH_4^+-N, VFAs, and TA.

Methods

Kinetic evaluation and calculation

The first-order kinetic model, Eq. 1, was used to determine the biogas production rate constant, whereas the modified Gompertz model, Eq. 2, was applied to determine the lag-phase and biogas production potential (Andriamanohiarisoamanana *et al.* 2017),

$$-ln(1-B/B_0)=kt \quad , \tag{1}$$

where B and B_0 (mL/g) are the cumulative and predicted biogas yields, which are equal to the interception obtained from plotting B against $1/t$, respectively, k (d^{-1}) is the biogas production rate constant, and t (d) is the digestion time (day),

$$C = C_0 \times \exp(-\exp\left[\frac{R_m \times e}{C_0}(\lambda - t) + 1\right]) \tag{2}$$

where C and C_0 (mL/g) are the cumulated biogas yield at time t and the potential biogas production, respectively, R_m (mL/(g TS.d)) is the maximum rate of biogas production, e = 2.71828, λ (d) is the lag time, and t (d) is the digestion time.

$$T_{90} = \lambda + 3.25 \times \frac{C_0}{R_m \times e} \tag{3}$$

$$T_{ef} = T_{90} - \lambda \tag{4}$$

In the above equations, T_{90} (d) is the period to generate 90% biogas yield, and T_{ef} (d) is the time period for effective biogas production (Andriamanohiarisoamanana *et al.* 2017).

The theoretical maximum yield of methane can be estimated according to the elementary composition,

$$C_cH_hO_oN_n + yH_2O \rightarrow xCH_4 + (c - x)CO_2 \tag{5}$$

where x=0.125(4c+h-2o-3n) is the theoretical maximum methane yield (TMMY).

Analytical methods

Daily biogas production was recorded on the basis of acidic saturated salt water displacement, and the value of standard temperature and pressure was noted (Liang *et al.* 2016). Methane content was determined by gas chromatography (Ruipu SP-6890; Ruihong, Lulan, China). Standard methods were used to measure the COD, NH_4^+-N, TS, and volatile solids (VS) (APHA 1995). The VFA was measured using the colorimetric ferric hydroxamate method, and the TA was determined by titrating a sample with standard hydrochloric acid to a pH of 4.3 (Liang *et al.* 2016). Elemental C, H, N, and S contents were determined using an elemental analyzer (Vario EL Cube; Elementar Analysensysteme GmbH, Langenselbold, Germany).

Statistical analysis

A one-way analysis of variance (ANOVA) and Fisher's least significant difference tests were performed to evaluate the data for any significant difference using SPSS 14.0 (SPSS Inc., Chicago, IL, USA) with a confidence interval of 95%.

RESULTS AND DISCUSSION

Moisture Content Influence on Biogas Production by BMP test

After 40 days of digestion, the specific biogas and methane yields of added VS ranged from 219.1 to 305.5 and 126 to 181.1 mL/g, respectively (Table 2 and Fig. S1). These methane yields were higher than that of the NaOH pretreatment of rose stalk at a TS concentration of 12.1% at 55 °C (Liang *et al.* 2016). After digestion for 40 days, the mean value for methane content ranged from 57.5% to 59.6% for four groups. A remarkable increase in biogas and methane productions occurred in the pretreatment groups compared with untreated group (Table 2), and the specific methane yield based on VS added increased 37% (R1), 40% (R2), and 44% (R3) compared with the CK group. Liang *et al.*

(2016) also reported that methane yield increased 8% to 44% with 1% to 4% NaOH pretreatment under an initial moisture of 52.3%. Moreover, a slight increase in specific biogas and methane yields was observed with the increase in moisture content of pretreatment from R1 to R3. The R3 group, with 77% moisture content during pretreatment, obtained the highest methane yield, but its yield was not significantly higher than those of the R1 and R2 groups. An increase in biogas yield with the increment of moisture content was also observed in the ammonia pretreatment of rice straw with 2% ammonia content (w/w) (Yuan *et al.* 2014). This phenomenon was ascribed to the increase in the alkali transfer rate and reaction extent, thereby resulting in a greater disruption of lignocellulosic structure, thus improving the digestibility of lignocellulosic biomass (Liang *et al.* 2011; Yuan *et al.* 2014).

Plotted experimental data and simulated values of kinetic equation are depicted in Fig. S2. Table 2 shows that the biogas production rate constant (k) of pretreatment groups ranged from 0.137 to 0.163 d^{-1}, which was higher than that of the CK group, and the highest k value was obtained for the R2 groups. Liotta *et al.* (2014) reported that an increase in k value was observed with the increment of moisture. The lag-phase (λ) ranged from 0 days to 0.8 days. The NaOH pretreatment significantly increased the biogas production potential (C_0) and the maximum rate of biogas production (R_m) compared with CK group. However, no significant difference in C_0 and R_m values was observed among the three pretreated moisture contents. Furthermore, a decrease in T_{90} and T_{ef} was observed for the pretreated biomass compared with the untreated biomass (Table 2). Besides, the methane production efficiency increased from 26.2% for CK to 35.7-37.8% for pretreated groups.

Table 2. Comparison for Biogas Production Efficiency and Kinetics of Anaerobic Digestion of Rose Stalk Under NaOH Pretreatment

Experimental Groups	CK	R1	R2	R3
Specific biogas yield (mL/g TS)	193.4 ± 28.5b	259.0 ± 20.9a	263.4 ± 7.3a	269.5 ± 26.3a
Specific biogas yield (mL/g VS)	219.1 ± 31.3b	293.6 ± 23.7a	298.5 ± 9.2a	305.5 ± 29.8a
Specific methane yield (mL/g TS)	111.2 ± 16.4b	151.8 ± 11.3a	155.9 ± 5.4a	160.6 ± 15.7a
Specific methane yield (mL/g VS)	126.0 ± 18.6b	173.2 ± 14.0a	176.7 ± 6.2a	181.1 ± 17.8a
Mean methane content (%)	57.5	59.0	59.2	59.6
TMMY (mL/g TS)	425.2	425.2	425.2	425.2
MPE (%)	26.2	35.7	36.7	37.8
k (d^{-1})	0.127 ± 0.018b	0.154 ± 0.012b	0.163 ± 0.023a	0.137 ± 0.019b
λ (d)	0.3 ± 0.3ab	0.0 ± 0.3b	0.8 ± 0.6a	0.4 ± 0.1ab
C_0 (mL/g TS)	212.6 ± 26.6b	279.4 ± 22.6a	287.0 ± 8.2a	288.5 ± 20.3a
R_m (mL/(g TS.d))	19.9 ± 1.5b	30.1 ± 2.4a	34.2 ± 8.01a	29.4 ± 2.8a
T_{90} (d)	13.1	11.1	10.0	12.1
T_{ef} (d)	12.8	11.1	10.8	11.7

TMMY, Theoretical maximum methane yield; MPE, Methane production efficiency, the ratio of Specific biogas yield (mL/g VS) and TMMY.
Note: the same row having the different letter are significantly different (p<0.05).

Among the studied groups, the R2 group, which had a pretreated moisture content of 70%, possessed a high methane yield, the highest k, C_0, and R_m values, and the lowest T_{90} and T_{ef} values. Thus, the 70% moisture content was selected for the solid-state NaOH pretreatment of rose stalk.

Anaerobic Codigestion Influence on Biogas Production

Biogas production from LBR experiment mainly occurred at the beginning of the 20th day of digestion. The maximum value of biogas production rate for CK (4621 mL/d), A1 (6435 mL/d), A2 (7862 mL/d), and A3 (7660 mL/d), occurred on days 4, 6, 4, and 4, respectively (Fig. 1A). After 40 days of digestion, the specific biogas and methane yields of added VS ranged from 213.2 to 336.7 and 145 to 221.2 mL/g, respectively (Fig. 1B and Table 3). The specific methane yield of the codigestion groups increased 41% to 52%, compared with the monodigestion of pretreated rose stalk. Marchetti *et al.* (2016) reported that the methane yield for the codigestion of wetland biomass and pig slurry was 30% higher than that of the monodigestion of wetland biomass. This phenomenon indicated that anaerobic codigestion enhanced methane yield due to the balanced essential nutrients, and fair C/N ratio (Andriamanohiarisoamanana *et al.* 2017). A1, A2 and A3 groups had a proper C/N ratio compared to CK group, as shown in Table 3. The A1 group, with a TS ratio of 1:1 for rose stalk and pig manure, obtained the highest specific biogas and methane yields. The mean methane content ranged from 66% to 68.2% for all groups (Table 3).

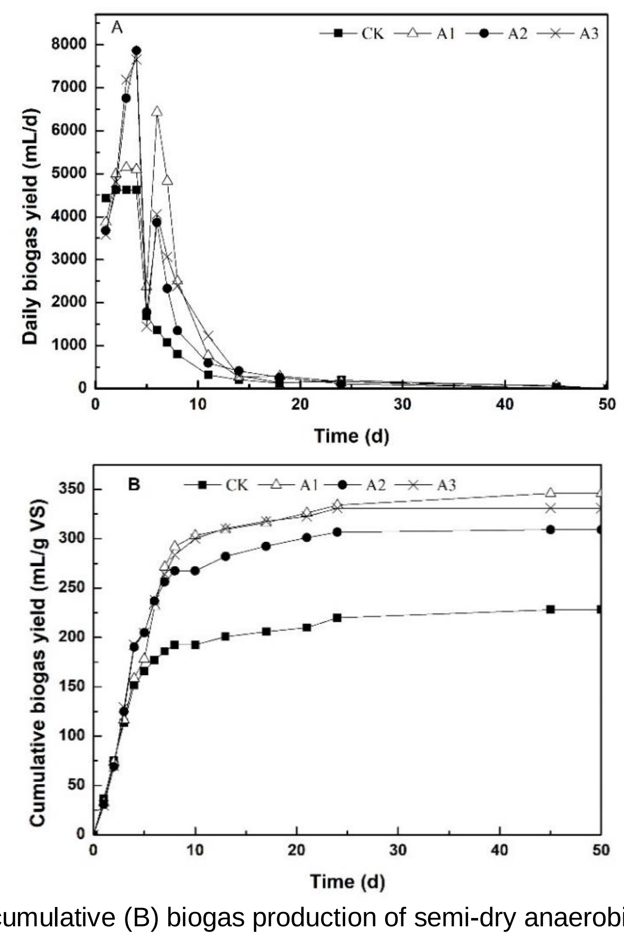

Fig. 1. Daily (A) and cumulative (B) biogas production of semi-dry anaerobic codigestion of rose stalk and pig manure with different TS ratios by LBR

By plotting experimental data and simulated values of kinetic equation is depicted in Fig. S3. Table 3 shows that the biogas production rate constant (*k*) of the CK group was higher than those of the A1 and A2 groups, but it was lower than that of A3. Moreover, the *k* value increased with the amount of pig manure. The λ values ranged from 0 days to 0.6 days, and the lowest λ value occurred in the CK group without pig manure. Codigestion of pretreated rose stalk with pig manure increased the biogas production potential (C_0) and the maximum rate of biogas production (R_m). Compared with the CK group, the R_m value of codigestion for pretreated rose stalk and pig manure increased 23% to 32%. Wu *et al.* (2010) found that the daily maximum biogas volume of anaerobic codigestion of swine and agricultural residues increased 6.12 to 11.4-fold compared with the monodigestion of swine manure. Furthermore, an increase in T_{90} and T_{ef} was observed for the codigestion groups (Table 3), which accorded with biogas production (Fig. 1B).

Table 3. Comparison for Biogas Production Efficiency and Kinetics of Anaerobic Codigestion of Rose Stalk and Pig Manure

Experimental Groups	CK	A1	A2	A3
Specific biogas yield (mL/g TS)	198.1	306.1	269.5	284.8
Specific biogas yield (mL/g VS)	213.2	336.7	298.5	316.5
Specific methane yield (mL/g TS)	134.7	201.0	185.0	194.3
Specific methane yield (mL/g VS)	145.0	221.2	204.9	215.9
Mean methane content (%)	68.0	66.0	68.7	68.2
TMMY (mL/g TS)	425.2	367.1	347.7	337.9
MPE	31.7	54.8	53.2	57.5
C/N ratio	29.4	21.8	19.3	18.0
k (d^{-1})	0.230	0.169	0.212	0.264
λ (d)	0.0	0.6	0.5	0.5
C_0 (mL/g TS)	213.4	331.7	300.0	325.8
R_m (mL/(g TS.d))	37.4	45.9	49.5	48.7
T_{90} (d)	6.8	9.2	7.7	8.5
T_{ef} (d)	6.8	8.6	7.2	8.5

TMMY, Theoretical maximum methane yield; MPE, Methane production efficiency, the ratio of Specific biogas yield (mL/g VS) and TMMY. The C/N ratio was calculated according to the C/N ratio raw sample of rose stalk and pig manure.

The above analysis shows that the anaerobic codigestion of pretreated rose stalk and pig manure remarkably increased the methane yield and changed the biogas production kinetics. The optimal performance for anaerobic codigestion was obtained at the TS ratio of rose stalk and pig manure of 1:1.

Anaerobic Codigestion Influence on Process Stability

Figure 2 presents the change in COD, VFA, NH_4^+-N, and TA contents of the leachate during anaerobic digestion. The COD contents ranged from 7 to 25 g/L for the four experimental groups (Fig. 2A), and high initial COD content (20 to 23 g/L) occurred in the CK, A1, and A2 groups. However, the peak value of COD happened on the 25th day of digestion for the A3 group. The reason for the late peak was the high COD generation during pretreatment and the high rate hydrolysis from the pretreated biomass.

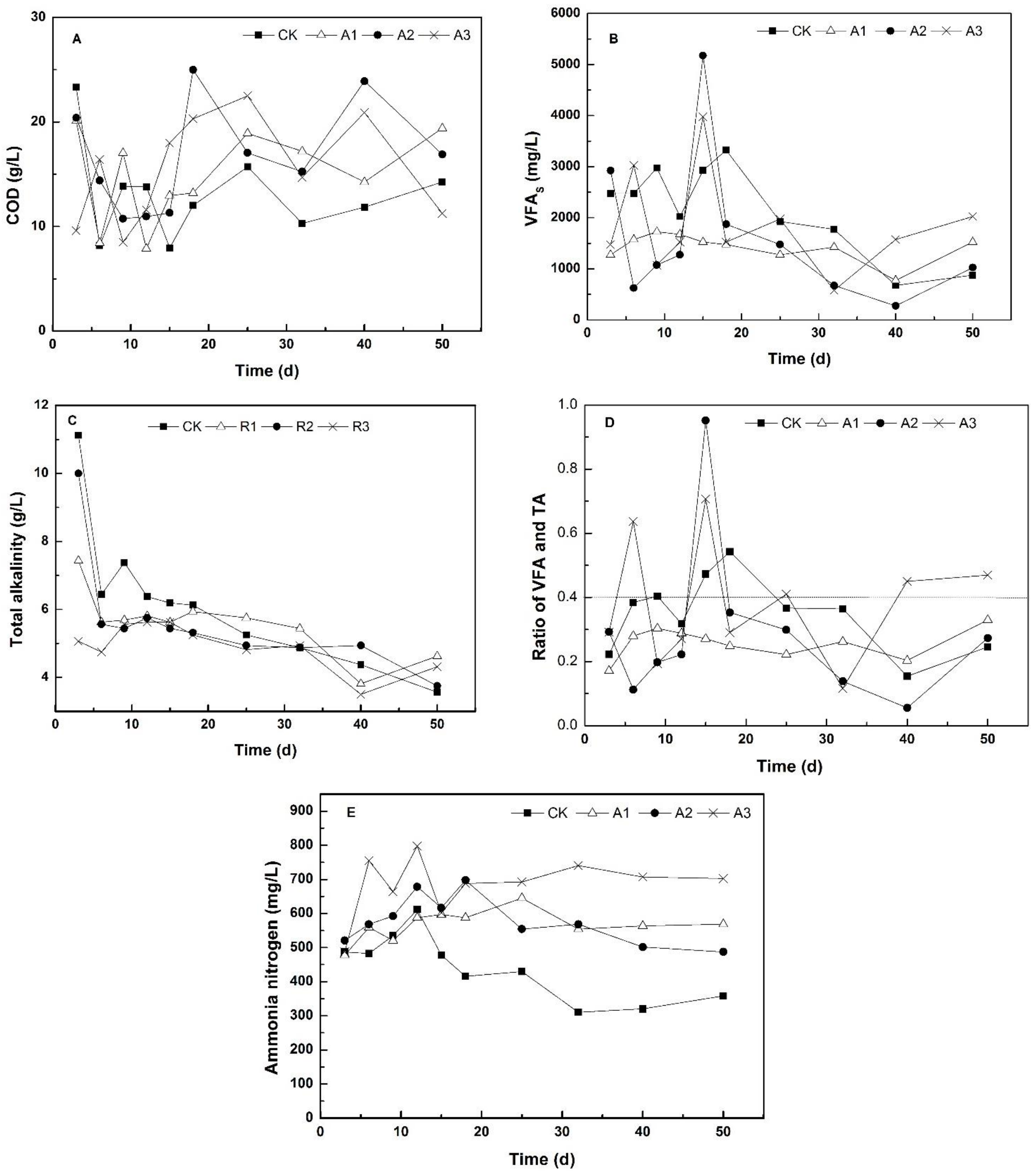

Fig. 3. Changes in COD (a), VFAs (b), TA (c), VFA/TA ratio (d), and NH4+-N (e) of LBR leachate from semi-dry anaerobic codigestion of rose stalk and pig manure

Liang *et al.* (2016) found that the COD value of pretreated liquid reached 41.3 g/L at a NaOH loading of 4% (w/w). No remarkable difference was found in the COD for the four experimental groups. The peak value of VFAs reached 2.9 g/L for CK (9th day), 1.7 g/L for A1 (9th day), 5.1 g/L for A2 (15th day), and 3.9 g/L for A3 (15th day), as shown in Fig. 2B. Siegert and Banks (2005) found that the inhibitory effect of VFAs on biogas production was evident above 6 g/L. This phenomenon suggested that VFAs could not inhibit biogas production. No remarkable difference was observed in the VFA contents of the four experimental groups, indicating that anaerobic codigestion did not greatly affect the VFA and COD values.

A high initial TA value followed by a rapid decrease occurred in the four experimental groups, because of the consumption of high pH value from pretreated rose stalk by initial VFAs (Fig. 2C). This phenomenon was also observed in dry anaerobic digestion after lime pretreatment (Zhang *et al.* 2016). The initial TA value decreased with the increase in pig manure. The VFA/TA ratio may be used as a stability indicator of the anaerobic digestion process, and a ratio below 0.4 indicates healthy digestion (Callaghan *et al.* 2002). The VFA/TA ratio at the digestion was partly higher than 0.4 for the four groups (Fig. 2D), implying a possible instability during the anaerobic digestion.

The presence of NH_4^+-N from pig manure decomposition tends to inhibit the performance of the anaerobic process. Its contents ranged from approximately 300 mg/L to 900 mg/L in the present study (Fig. 2E). This finding suggested that no inhibition from NH_4^+-N occurred during the anaerobic digestion of pig manure because Rajagopal *et al.* (2013) reported that 200 to 1000 mg/L of NH_4^+-N had no antagonistic effect on anaerobic digestion. The addition of pig manure remarkably increased NH_4^+-N content compared with CK group, and the NH_4^+-N of A3 group was remarkably higher than other groups.

CONCLUSIONS

1. Solid-state NaOH pretreatment increased methane yield 37% to 44%, along with biogas production kinetics.
2. No significant difference in methane production and biogas production kinetics was observed at three moisture contents (54%, 70%, and 77%) .
3. The anaerobic codigestion of rose stalk and pig manure increased 41% to 52% of methane yields and improved biogas production kinetics compared with monodigestion of rose stalk. The optimal performance for anaerobic codigestion was obtained at the TS ratio of rose stalk to pig manure of 1:1.

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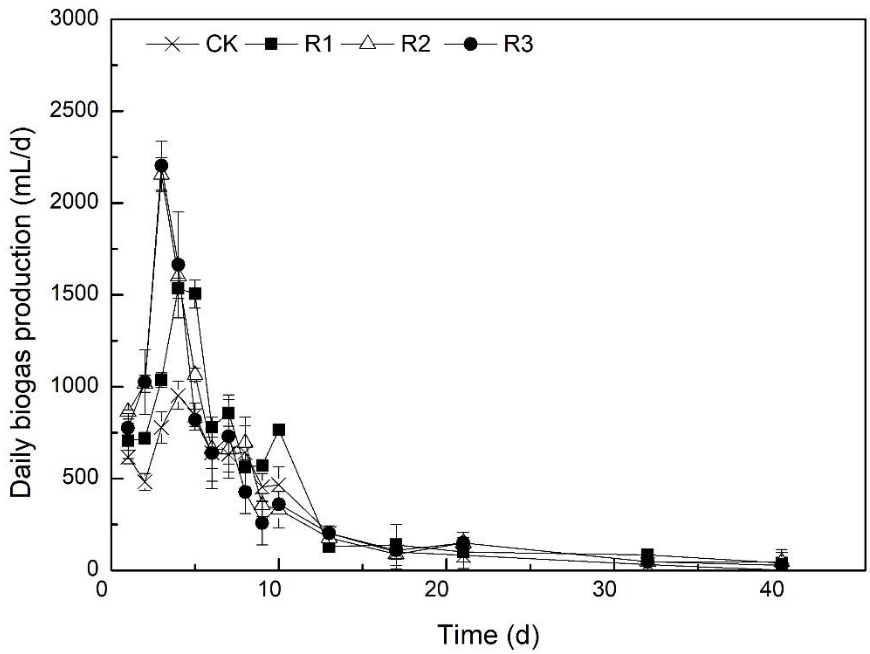
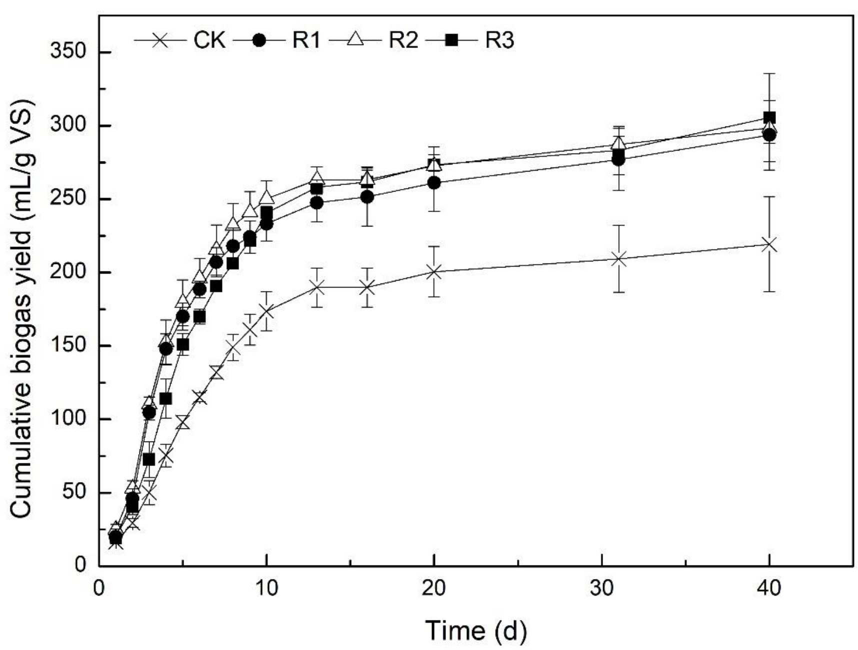
APPENDIX

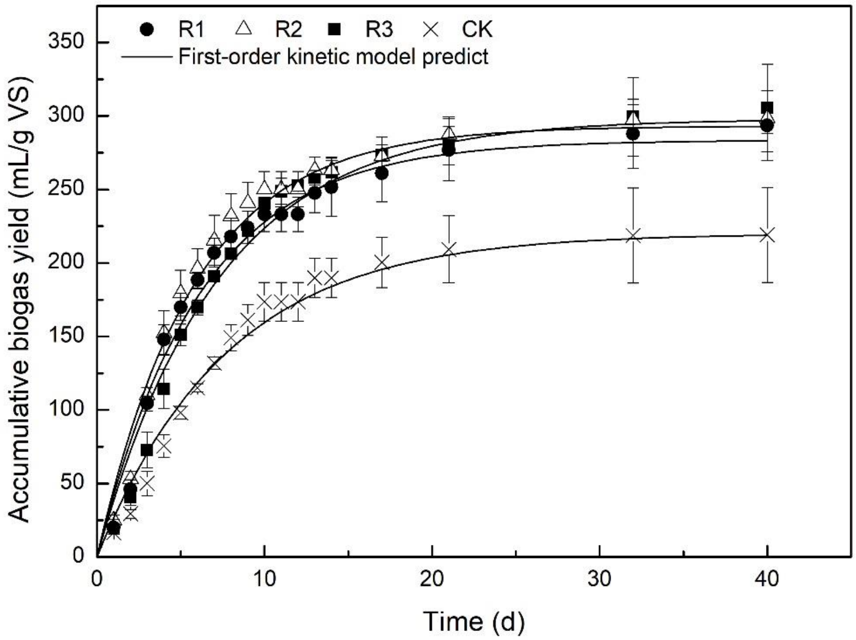


a



b

Fig. S1. Daily (a) and cumulative (b) biogas production of semi-dry anaerobic digestion of rose stalk under solid-state NaOH pretreatment with different moisture content



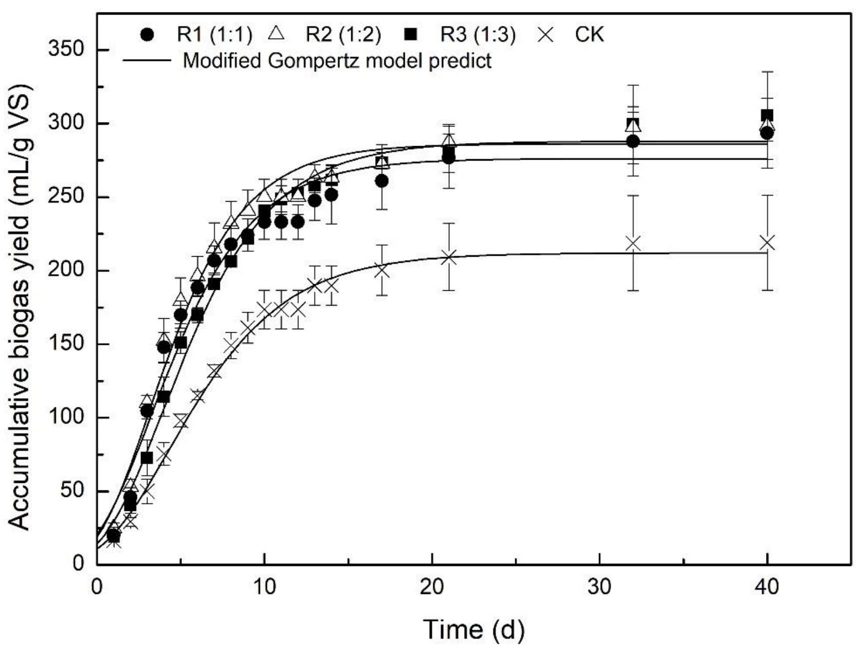
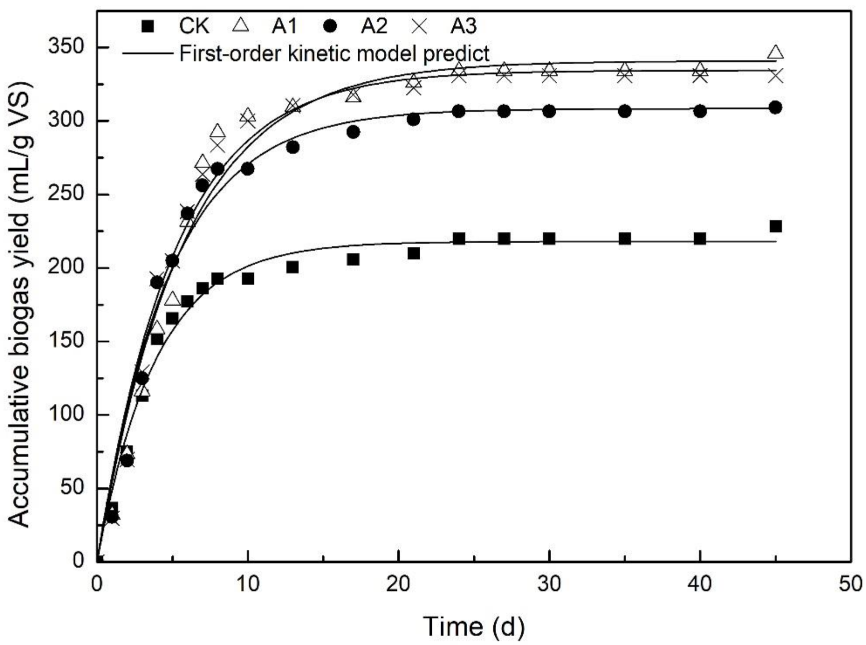


Fig. S2. Variation of experimental data and calculated value of cumulative biogas production of semi-dry anaerobic digestion of rose stalk under solid-state NaOH pretreatment



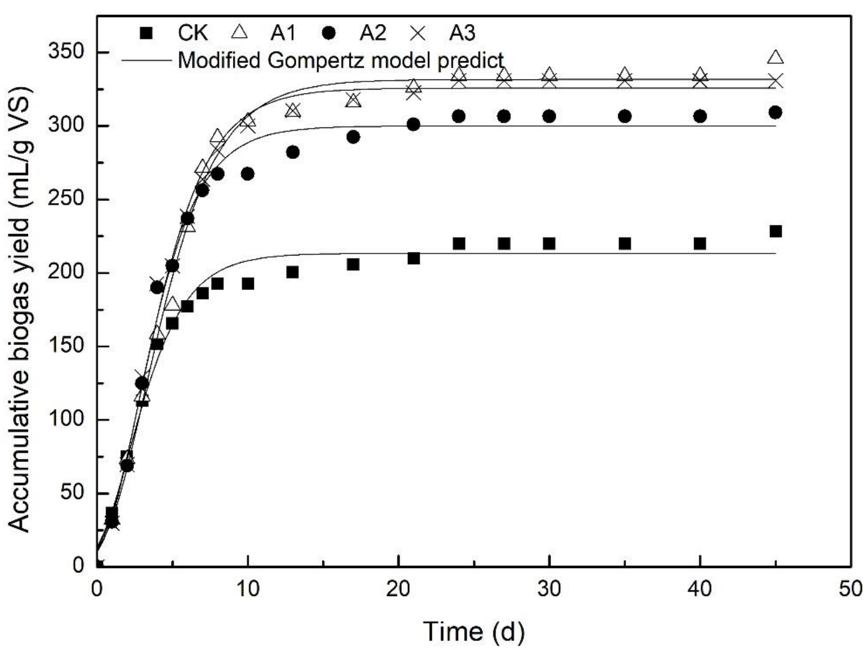


Fig. S3. Variation of experimental data and calculated value of cumulative biogas production of semi-dry anaerobic codigestion