Desalination and Water Treatment
www.deswater.com
doi: 10.5004/dwt.2020.26077

198 (2020) 307–313
September

Methane production kinetics in the combination of high temperature thermal hydrolysis pretreatment and anaerobic co-digestion for municipal sludge and banana straw

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Received 16 August 2019; Accepted 6 May 2020

ABSTRACT

The anaerobic digestion (AD) of municipal sludge (MS) become a popular way for the treatment of municipal sludge. In this study, anaerobic co-digestion reactors were applied for the digestion of municipal sludge together with banana straw (BS). The biochemical methane potential in the AD system of municipal sludge, banana straw, and their mixture was modeled by first-order kinetics. The methane yield of the mixed substrates (MS + BS) was significantly increased compared to the digestion of a single substrate (MS or BS). Additionally, the co-digestion reactors were combined with high-temperature thermal hydrolysis pretreatment. In the combining system, the methane yields of municipal sludge, banana straw, and their mixture were 388, 372, and 537 mL g^{-1} VS (VS – volatile solids), respectively. The calculated methane yield value (380 mL g^{-1} VS) in the combining system with the mixed substrates (MS + BS) is significantly lower than the measured value (537 mL g^{-1} VS), indicating a significant synergistic effect of municipal sludge and banana straw during the digestion. The methane production of the pretreated substrates (MS, MS + BS) followed first-order kinetics except for the pretreated banana straw. However, the first-order kinetic cannot precisely predict the methane productions of raw municipal sludge, raw banana straw, mainly due to the low hydrolysis ability. Additionally, the volatile fatty acids/total alkalinity value of all the experiments, except the pretreated banana straw, suggested stable digestion systems. In the co-digestion system with pretreated mixed substrates (MS + BS), the C/N ratio (26.4) and high degradation ratio of VS (59.2%) resulted in the high methane yield.

Keywords: Municipal sludge; Banana straw; Biochemical methane potential; Anaerobic co-digestion; Kinetics

1. Introduction

Municipal sludge (MS) comes out as an unexpected by-product in the sewage treatment plants. The production of the MS with 20% total solid in urban areas of China, according to the statistics from the Ministry of Housing and Urban-Rural Development, has reached 40 million tons [1]. Inappropriate treatment and disposal of these MS bring secondary pollution to the environment. As such, in recent years one technology called anaerobic digestion (AD) of MS has been widely used in northern China. Another pretreatment known as high-temperature thermal hydrolysis

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pretreatment (HTHP) is applied in several studies for the purpose of improving biodegradability and enhancing dewaterability [2,3]. However, the relatively low organic matter content (<50%) leads to low methane productions, which restricts its popularization in southern China [4].

The banana plant is a large annually harvested herbaceous flowering plant. A study reported that the production of 1-ton bananas brings about 2.4 tons of straws [5]. In 2015, the banana straw (BS) in China was around 30.34 million tons. This BS, mainly from southern China, are discarded in the field scarcely with any treatment. This discarding of BS leads to the growth of massive molds emitting rotten smell in hot and humid weather, which also causes severe contamination [6].

Anaerobic co-digestion is widely used in sewage treatment plants owing to its strong synergistic function of the simultaneous degradation of organic wastes [7]. The co-digestion process integrates the digestions of two or more substrates, which not only increases the methane yield but also alleviates adverse effects. One well-studied co-digestion process is the synergistic ADs of sludge and kitchen waste. This is attributed to the fact that the organic compounds matter in kitchen waste can significantly improve the stability of the system and enhance biogas production [8]. Thereby, BS, with high organic composition as kitchen waste, is also potential objects for anaerobic co-digestion with MS.

To the best of our knowledge, the existing studies were only focused on the HTHP of MS [2,3], while the HTHP of BS, especially the co-digestion of MS and BS both pretreated by HTHP, has not been investigated. Therefore, due to its high potential applications, it is interesting to investigate the methane production kinetics during the MS and BS anaerobic co-digestion coupled with HTHP, from both modeling and experimental perspectives. As to the modeling part, it is well known that the Gompertz model [9,10] and first-order kinetic [11] were widely used for the evaluation of methane production kinetics during AD. The Gompertz model is suitable for AD of raw materials with lag time during methane production. The first-order kinetic model is more suitable for AD of raw materials without lag time during methane production [12].

This study aimed to characterize the methane production and its kinetics during the anaerobic co-digestion of MS and BS coupled with HTHP. Different sets of AD experiments were conducted for two groups: (1) pretreated (HTHP) MS, BS, and their mixture, and (2) raw MS, BS, and their mixture. Different experiment sets were compared on the methane production kinetics.

2. Materials and methods

2.1. Experimental materials

The MS (total solid TS = 17.8%, volatile solid VS = 8.5%) used in the study was the dewatered sludge from a municipal sewage treatment plant in Nanning, Guangxi, China. The BS (TS = 89.0%, VS = 81.4%, cellulose = 37.9%, hemicelluloses = 21.9%, lignin = 17.0%) was provided by Tanluo banana farm in Nanning. The BS was dried and crushed to a size less than 10mm, and then stored in a plastic bag before using. Sludge for inoculation (TS = 14.2%, VS = 9.6%) was collected from the granular sludge of an internal circulation (IC) reactor. The inoculum was pre-incubated in the water bath (35°C ± 1°C) for 20 d to deplete any residual biodegradable organics.

2.2. High-temperature thermal hydrolysis pretreatment

The HTHP of the MS was conducted in a 1 L electric-heating reactor (SZCL-2, TianHeng Instruments, China). The optimal temperature of HTHP for MS, BS, and their mixture (1:1, VS:VS) are 170°C, 190°C, and 190°C, respectively, and the optimal hold time for 30 min for all groups [13]. Thermal pretreated sludge was stored at 4°C for no more than 5 d before further analysis and tests.

2.3. Anaerobic digestion

The biochemical methane potential (BMP) tests allow determination of the ultimate methane potential yield for several solid substrates by AD in specific conditions [14,15]. In this study, the tests were conducted in serum bottles (250 mL), which were flushed with nitrogen for 5 min. The working volume was 150 mL after inoculation and the spiking of the substrate. The reactors were sealed with rubber plugs and were placed in HH-S digital constant temperature water bath at mesophilic conditions (35°C ± 1°C). During the experiment, the reactors were stirred in a horizontal shaker. The substrate to the inoculum ratio of all the reactors was set to 1:1 VS. The TS concentration and the VS ratios of MS/BS were 10% and 1:1, respectively. For the MS system, the TS of MS and inoculum were 8.8 and 6.2 g, respectively. For the BS system, the TS of BS and inoculum were 6.4 and 8.6 g, respectively. For the MS and BS mixture system, the TS of MS, BS, and inoculum was 5.1, 2.7, and 7.2 g, respectively. Sample without pretreatment was set as the control. The methane productions were measured with the liquid displacement method by using a sodium hydroxide solution (NaOH, 5%) to absorb the acid gases such as CO_2 and H_2S completely [16,17]. The measurement of methane production for the groups with and without HTHP was stopped almost at the same time no more methane was produced. Each BMP test was conducted in triplicate and the arithmetic average of each parameter was taken as the final data.

2.4. Analysis methods

In order to avoid the biomass losses of the system, samples were only taken from the raw materials, thermal hydrolysis products, and AD effluent. By centrifugation at 5,000 rpm for 10 min, the supernatant was filtered for analyzing total alkalinity (TA), pH, and volatile fatty acids (VFA). The pellet, separating from the supernatant after centrifuge, was used for analyzing TS, VS, total carbon, total carbon, cellulose, hemicellulose, and lignin. The samples were analyzed by the following methods VFA was determined by the colorimetric method [18]. TA was titrated by a bromocresol green-methyl red indicator [19]. C/N ratio was calculated according to the total carbon and total nitrogen measured from the all-element automatic analyzer (EA3000); STARTER2100 precise pH meter was

adopted for pH; weight was measured by TS and VS [20]. VAN SOEST method was used for cellulose, hemicellulose, and lignin [21].

2.5. Calculation

The first-order kinetic model was applied to evaluate the cumulative methane yield of the raw materials, stated as Eq. (1).

$$Q_t = Q_e \cdot \left[1 - \exp\left(-k_h \cdot t\right)\right] \tag{1}$$

where Q_t the cumulative methane yield, mL g^{-1} (VS); Q_e the maximum methane yield, mL g^{-1} (VS); k_h first-order hydrolysis coefficient, d^{-1}; t the digestion time, d; E the Euler constant, equal to 2.718, and Q_e, k_h can be obtained by the fitting in Origin 9.0.

The cumulative methane yield of the mixed substrates can be calculated according to Eq. (2):

$$Q = r \times Q_{t\mathrm{MS}} + (1 - r) \times Q_{t\mathrm{BS}} \tag{2}$$

where Q cumulative methane yield from mixed substrates, mL g^{-1} VS; r the ratio of thermally hydrolyzed sludge in the mixed substrate (VS/VS); $Q_{t\mathrm{MS}}$ cumulative methane yield from thermal hydrolysis MS, mL g^{-1} VS; $Q_{t\mathrm{BS}}$ cumulative methane yield from thermal hydrolysis BS, mL g^{-1} VS.

The synergistic effects of an anaerobic mixed matrix are reflected in the relative deviation of the actual cumulative methane production curve and the theoretical value, which can be expressed by the relative errors of the two within the fermentation cycle as shown in Eq. (3):

$$\mathrm{RD} = \frac{\left(Q_{t\mathrm{MS}} - Q\right)}{Q} \times 100\% \tag{3}$$

where RD relative deviation degree, %; $Q_{t\mathrm{MS}}$ the experimental value of cumulative methane production, mL g^{-1} VS; Q the fitting value of cumulative methane production, mL g^{-1} VS.

The deviation between the fitting value and the measured value of methane yield can be calculated by Eqs. (1) and (3).

3. Results and discussion

3.1. Methane yield

The daily methane productions are shown in Figs. 1a–c. It shows that the reactor with the HTHP of MS, BS, and their mixture had much higher methane production than those of the experiments without HTHP on the first day. This indicates that the HTHP can significantly increase methane production with these different types of substrates. However, it is also apparent that the effect of HTHP was quite different for the three substrates by noting that as the methane production on the first day showed significant variations from 12 to 60 ml (g VS d)$^{-1}$. The reactor with the mixture had the maximal daily methane production with 60 ml (g VS d)$^{-1}$ on the first day. Later it was stable

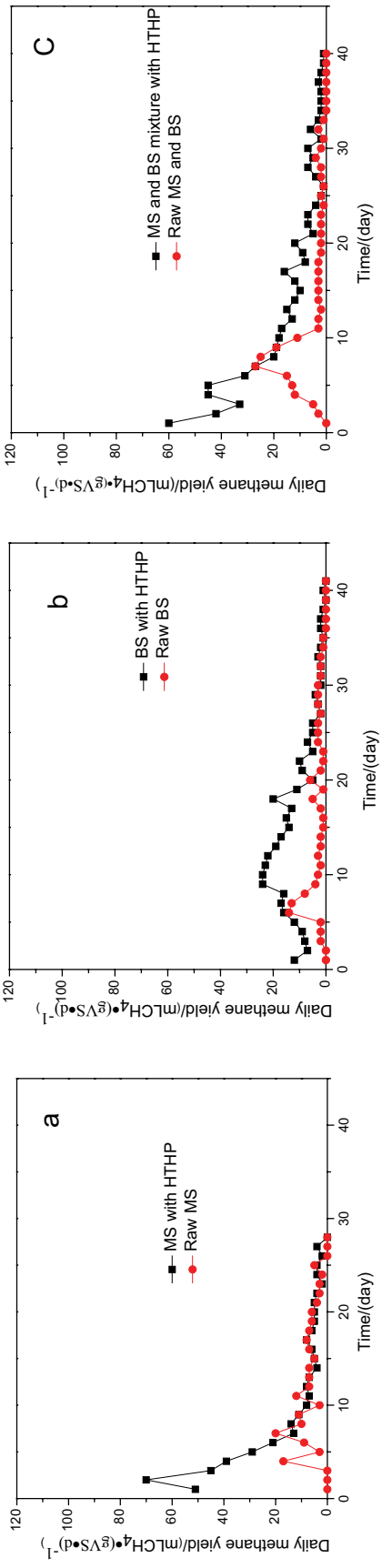

Fig. 1. Daily methane yield of AD with different substrates.

at around 30 ml (g VS d)$^{-1}$ for 5 d, and then decreased gradually. The pretreated MS had a high volume in the first 2 d with a peak value of 70 ml (g^{-1} VS d) on the second day. The daily methane production in the group with thermal hydrolyzed BS increased in the first 10 d and decreased later. The single daily methane production was about 24 ml (g^{-1} VS d).

The cumulative methane yields of pretreated MS, BS, and their mixture are shown in Fig. 2. All the cumulative methane yields in the group with HTHP were more than 300 mL g^{-1} VS, and in contrast, all the cumulative methane yields in the group without HTHP were less than 200 mL g^{-1} VS. This result suggests that HTHP significantly increases methane production, which is consistent with the existed studies [12,22]. The group of raw MS and mixture generated almost the same methane accumulation. However, in the group with HTHP, the reactor with the mixture had much more methane production indicating that HTHP differently affected MS and mixture. Similarly, the methane production of the group with raw BS was different from the group with raw MS. However, almost the same methane accumulations were obtained for MS and BS with HTHP. This also indicates that the effect of HTHP for the reactor with BS was different from MS. In the experiments with HTHP, the methane productions in the reactor with MS, BS, and mixture were 388, 372, and 537 mL g^{-1} VS. With Eq. (2), the Q value calculated from Q_{MS} and Q_{BS} is 380 mL g^{-1} VS. The calculated Q value is significantly lower than the real measured value (537 mL g^{-1} VS), which indicates a significant synergistic function of MS and BS. The cumulative methane production of the mixture in the first 3 d exceeded the total gas production of the control. The technical digestion time (T80, the time required to produce 80% of total biogas production), as an important parameter, can be a reference for designing hydraulic residence time (HRT). The biogas production based on T80 can reflect the final biogas production in engineering applications [23,24]. In the group with HTHP, the T80 of the reactors with MS, BS, and the mixture were 12, 19, and 17 d, respectively. These values can be considered as the HRT for application. In the group without HTHP, the T80 were 18 (MS), 26 (BS), and 17 (mixture). Obviously, the T80 of the group without HTHP were all relatively longer than the group with HTHP, which indicates the pretreatment can reduce the HRT during the application.

The synergistic effect of pretreated MS and BS for methane production in AD can be quantified by Eqs. (2) and (3). The relative deviation was reached to 90.5% on the first day, which indicates a significant synergistic effect of the mixture. This also indicates that the dissolved organic matter of the substrates increased rapidly after HTHP, which is consistent with previous studies. For example, Li and Noike [25] reported that HTHP (30–60 min at 170°C) significantly increased methane yield by 2 times, as compared with the control. Cano et al. [26] found thermal hydrolysis has led to an increase of the methane productions (more than 50%) and kinetics parameters (even double). In the pretreatment, for MS, due to the cell lysis that took place and especially in the steam explosion, the cell wall was ruptured. Additionally, complex organic matters (proteins, carbohydrates, lipids, etc.) were transformed into simple soluble matters (amino acids, sugars, fatty acids, etc.), which enhances the hydrolysis rate and increases the methane production. In the case of BS, the long-chain cellulose and lignin were cut off after HTHP, which significantly improved the biodegradability [27]. Moreover, co-digestion with BS could improve the nutrient balance and biodegradability due to the high VS (80%–90%) and C/N (40–45). By calculation, combined with the synergistic anaerobic co-digestion of the MS and BS, the carbon/nitrogen ratio in the reactors changed to 26.4 which was suitable for the growth of microbes and led to the increase of methane production rate. Previous studies have reported that the optimal proportion range for anaerobic bacteria growth is from 20 to 30 [28,29], which significantly affected the biogas yield confirmed in this study.

3.2. Methane production kinetics

First-order kinetics was used to predict the methane yield of MS, BS, and their mixture in all the groups. The fitting methane yield and hydrolysis constant of each group in the pretreatment are summarized in Table 1. Kinetic fitting of methane yields during AD of MS, BS, and their mixture are presented in Fig. 2. The methane production in the reactor with pretreated MS followed the first-order kinetic (R^2 = 0.983) with the predicted methane yield of 375 ± 4 mL g^{-1} VS which is only 13 mL g^{-1} VS lower than the measured value. Further, the hydrolysis constant was 0.18 ± 0.007, which indicates the pretreated MS was not affected by the lag phase. However, the predicted methane production of raw MS was 400 ± 104 mL g^{-1} VS, which was 237 mL g^{-1} VS higher than the measured value. This discrepancy suggests the first-order kinetic cannot accurately predict the methane production of raw MS. The relatively low hydrolysis constant (0.02 ± 0.007) also indicates that hydrolysis was the limitation in the reactor.

In the reactor with pretreated BS, the predicted methane yield was 479 ± 27 mL g^{-1} VS, which was 102 mL g^{-1} VS higher than the actual measured value. This discrepancy means the first-order kinetic also cannot accurately predict

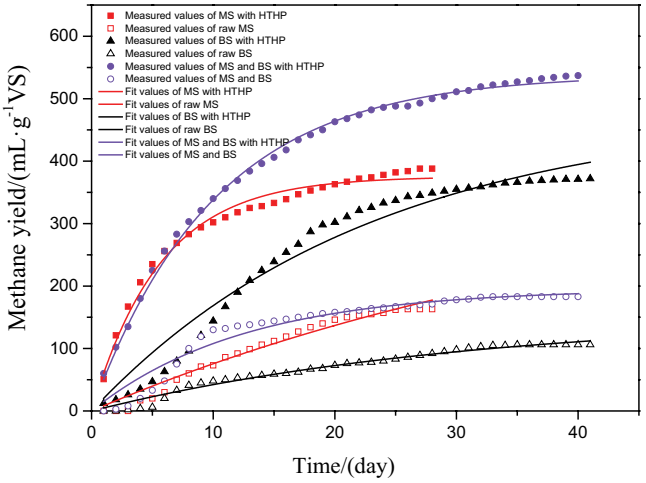

Fig. 2. Accumulations and Kinetic fitting of methane yield during AD of the different substrates.

the methane production of pretreated BS. Similarly, as in the reactor with raw MS, the low hydrolysis constant (0.04 ± 0.005) was obtained, which also indicates hydrolysis was the limitation for methane production. After AD, the compositions of cellulose and lignin, as the indicator for the residual non-biodegradable substances, decreased to 10.3% and 6.5%, respectively. This is consistent with the existing study [30] that straw substances have a lag time in methane production, and long-chain cellulose showed a lower degradation. To the raw BS, the hydrolysis constant was only 0.03, indicating the restriction by hydrolysis.

After HTHP, the methane production kinetics in the reactor with MS and BS mixture agreed with the first-order kinetics (R^2 = 0.997) with the predicted methane yield of 539 ± 3 mL g^{-1} VS which was only 2 mL g^{-1} VS less than the accumulated methane. The compositions of cellulose and lignin were 6.2% and 3.5%, respectively, indicating the good bio-availability of the mixture. Further, the corresponding hydrolysis constant was 0.10 ± 0.001, indicating that no lag phase existed, and the first-order kinetics fit the gas generation well. According to the kinetic parameters of the raw mixture, the corresponding hydrolysis constant was 0.08, which was three to four times of the single substrate (raw MS or BS). To some extent, the synergism of the two groups reduced the influence of hydrolysis restriction.

3.3. Physicochemical indexes in the reactors

The physicochemical indexes of each reaction group are summarized in Table 2. The TA and ammonia nitrogen (NH_3–N) increased from 2,660 ± 35 to 18,950 ± 42 mg L^{-1} and from 1,020 ± 15 mg L^{-1} to 2830 ± 23 mg L^{-1}, respectively. As summarized by Chen et al. [31], a wide range of inhibiting ammonia concentrations has been reported in the literature, with the inhibitory total ammonia nitrogen concentration that caused a 50% reduction in methane production ranging from 1.7 to 14 g L^{-1}. In our study, only the NH_3–N concentration of the group with pretreated mixed substrates (MS and BS) was in the range, which potentially indicates the inhibitory. The VFA in the anaerobic co-digestion reactors also accumulated significantly (from 810 ± 12 to 4,100 ± 25 mg L^{-1}), which almost in the tolerance concentrations (4,000–8,000 mg L^{-1}) as reported previously [32]. Another parameter, the VFA/TA value was considered as one of the indicators for the stability of the AD system (<0.4) [33]. In our study, the VFA/TA value of the reactor with the pretreated mixed substrates (MS and BS) was 0.218, which indicates the stable state of the anaerobic system with a high amount of methane production. The higher VFA/TA value of the reactors with pretreated BS (0.551), which were out of the stable range (<0.4), can be the cause reason for the lower methane yield compared to the reactors with pretreated MS and BS mixture. To the best of our knowledge, a higher the degradation rate of VS and soluble chemical oxygen demand (sCOD), accompanying with the higher accumulation of VFA concentration, leads to higher methane yield. In the reactor with pretreated MS and BS mixture, with the maximum methane production, the removal rate of sCOD and VS were 94.5% and 59.2%, respectively, which are higher than these of the reactors with pretreated MS (93.7% and 48.4%) or BS (89.8% and 48.8%).

4. Conclusion

- With HTHP, the methane yields of MS, BS, and their mixture were 388, 372, and 537 mL g^{-1} VS, respectively. The calculated methane yield value in the combining

Table 1
Kinetic parameters of AD of MS, BS and their mixture

Parameters \ Group	MS		BS		MS and BS	
	With HTHP	Raw	With HTHP	Raw	With HTHP	Raw
Q_e / (mL g^{-1} VS)	375 ± 4	400 ± 104	479 ± 27	153 ± 10	539 ± 3	196 ± 6
k_h /d^{-1}	0.18 ± 0.007	0.02 ± 0.007	0.04 ± 0.005	0.03 ± 0.004	0.10 ± 0.001	0.08 ± 0.007
Adj. R^2	0.983	0.968	0.962	0.974	0.997	0.943

Table 2
Physicochemical characteristics of experimental materials

Group	pH	TA/mg L^{-1}	VFA /mg L^{-1}	VFA/TA	sCOD/mg L^{-1}	NH_3–N/mg L^{-1}	VS/%
Raw MS	7.0 ± 0.2	1,400 ± 23	213 ± 15	0.152	618 ± 20	403 ± 17	5.60 ± 0.003
MS after HTHP	6.7 ± 0.3	9,730 ± 30	2,180 ± 42	0.224	8,900 ± 20	1,266 ± 15	–
MS after AD	–	–	–	–	565 ± 30	–	2.89 ± 0.004
Raw BS	–	–	580 ± 15	–	2,045 ± 20	–	7.80 ± 0.004
BS after HTHP	6.5 ± 0.1	2,950	1,625 ± 26	0.551	5,725 ± 25	652 ± 20	–
BS after AD	–	–	103 ± 13	–	586 ± 16	–	3.99 ± 0.004
Raw MS and BS	7.2 ± 0.2	2,660 ± 35	810 ± 12	0.305	1,893 ± 20	1,020 ± 15	6.54 ± 0.003
MS and BS after HTHP	6.9 ± 0.3	18,950 ± 42	4,100 ± 25	0.218	11,126 ± 43	2,830 ± 23	–
MS and BS after AD	–	–	–	–	608 ± 16	–	2.67 ± 0.004

system with the mixed substrates is significantly lower than the measured value, indicating a significant synergistic effect of MS and BS.

- The results showed that, with HTHP, the two methane yields of the MS and its mixture with BS had 375 and 539 mL g^{-1} VS, respectively, consistent with the prediction of the first-order kinetics. However, the first-order kinetic cannot accurately predict the methane production of raw MS, raw BS, and pretreated BS, and their hydrolysis constants value also showed a relatively long lag phase. The synergism of the raw MS and BS reduced the influence of hydrolysis restriction.
- In this study, except for the BS after HTHP, other groups showed stable states. However, the methane yield in AD for MS with HTHP was also less than the pretreated MS and BS mixture due to the low C/N ratio. The biodegradation of mixed substrates had a significant synergistic effect, while both the methane yield (537 mL g^{-1} VS) and the removal rate of VS (59.2%) are higher than MS (388 mL g^{-1} VS, 48.4%) and BS (372 mL g^{-1} VS, 48.8%) alone, which suggests the methane yield is positively correlated to the degradation rate of VS.

Acknowledgment

This work was supported by the National Natural Science Foundation of China (No. 21167003) and the Natural Science Foundation of Guangxi (No. 2016GXNSFAA380294).

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