Chemical Engineering Journal 264 (2015) 174–180

Contents lists available at ScienceDirect

Chemical Engineering Journal

journal homepage: www.elsevier.com/locate/cej

Effects of thermal hydrolysis on organic matter solubilization and anaerobic digestion of high solid sludge

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HIGHLIGHTS

- THP positively affected the organics solubilization of high-solid sludge.
- THP of high solid sludge greatly determined the performance of anaerobic digestion.
- HTHP significantly sped up the gas production rate and increased the total gas volume.
- The SRT could be reduced after the thermal hydrolysis at 140–160 °C.

ARTICLE INFO

Article history:
Received 10 June 2014
Received in revised form 21 October 2014
Accepted 1 November 2014
Available online 13 November 2014

Keywords:
Anaerobic digestion
High solid sludge
Thermal hydrolysis process
Organic matter solubilization
Sludge viscosity

ABSTRACT

Anaerobic digestion of sludge not only decreases the mass/volume of sludge, but also increases the production of energy-rich biogas. The effects of a thermal hydrolysis process (THP) on the solubilization of main organics of sludge, as well as the performance of the followed biochemical methane potential (BMP) tests under mesophilic condition (35 °C), were systematically evaluated. Organic compounds such as proteins and carbohydrates which have relatively high yield coefficients are solubilized efficiently during the THP. Good linear relations were obtained between the amounts of soluble chemical oxygen demand and soluble proteins/carbohydrates. The viscosity of sludge dramatically reduced after the THP, particularly at higher temperatures. In contrast to the treatments by low-temperature THP (60–90 °C), the treatments by high-temperature THP (120–160 °C) accelerated the digestion rate and increased the biogas yield in the following BMP tests. Meanwhile, the solid retention time could be reduced from 18–20 days to 12–14 days when high solid samples were pre-treated under THP at 140–160 °C based on the corresponding results of acceleration of methane production after the treatment. As a result, this study elucidated the appropriate operational conditions for high solid sludge (TS > 10%) under THP, and their mechanisms of the acceleration of biogas production.

1. Introduction

Anaerobic digestion is applied popularly to sewage sludge stabilization. It has many advantages such as reduced mass/volume of the sludge, production of energy-rich biogas, and improved sludge dewaterability [1]. Moreover, compared to aerobic digestion, anaerobic digestion costs less, and need smaller environmental footprints [2]. Anaerobic digestion includes four major steps: hydrolysis, acidogenesis, acetogenesis, and methanogenesis. In the first step, hydrolysis, both the solubilization of particulate matter and biological decomposition of organic polymers to monomers/dimers occur slowly, thus making it the rate-limiting step of the overall processes [12].

When it comes to the hydrolysis of sludge integrations, the release of intracellular and extracellular matters becomes the most important. Thermal pretreatment processes thus have been studied to promote sludge hydrolysis by destroying the integrations, which results in the reduction of solid retention time in digestion process by finally accelerating methane production. In addition, the sludge after enhanced hygienization of thermal processes can not only comply with the European Union (EU) policy on the elimination of pathogens [10], but also reach class A bio-solid standard of the US Environmental Protection Agency (EPA) [15], making it more suitable for subsequent land application.

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http://dx.doi.org/10.1016/j.cej.2014.11.005
1385-8947/

Since the pretreatment under high temperature (>100 °C) and high pressure (10 M) consumes more energy and requires strictly for the equipments, pretreatment under low temperature for organic matters has attracted much attention recently. Wang et al. [16] tested the effectiveness of low-temperature pretreatment (60–100 °C) on the mesophilic anaerobic digestion of sludge and found that it significantly increased (30–52%) the methane production. The generation rate of methane production sped up more after the pretreatment at 60 °C compared to that observed for both at 80 °C and 100 °C. However, the thermal pretreatment time adopted for all the experiments in the study was only 30 min; the superiority of higher temperature might not have been well elucidated. Climent et al. [6] reported that the optimal pretreatment for secondary sludge with 70 °C was for 9 h rather than for 24 h, 48 h, and 72 h. Meanwhile, different raw sludge and/or different pretreatment conditions might lead to inconsistent or even contrary results [16,4]. And most studies were carried out either at one temperature for different pretreatment times or over a narrow temperature range for a determined pretreatment time, and sometimes the results also led to the contrary conclusions, it is necessary to conduct a systematical analysis of thermal hydrolysis over a broader range of temperature and pretreatment time using same sludge, thereby to comprehensively elaborate the effects of THP on organic matter solubilization of sludge and the performance of the following anaerobic digestion.

Once the temperature exceeds 200 °C during the thermal-pressurized pretreatment, the Maillard reactions will happen, by which melanoidins will be produced in the reactions between reducing sugars and amino acids. However, melanoidins are high-molecular-weight heterogeneous polymers that are difficult to degrade or even inhibit the degradation of other organics [9]. Abe et al. [1] reported that biogas production by anaerobic digestion of sludge with thermal pretreatment at 200 °C decreased by 33% in comparison to the gas yield from the digestion of sludge with thermal pretreatment at 170 °C, and even less than the control in which the fed sludge was not pretreated.

Moreover, the concentrations of total solids (TS) in sludge in the previous studies were mainly 3–10%, and dewatered sludge with TS ≈ 20% had been rarely studied. In comparison to low solid anaerobic digestion, high solid anaerobic digestion (TS > 10%) is more attractive because of the relatively smaller reactor volumes, lower energy requirements for heating, less material handling, etc [13]. Nowadays, centralized processing of collected dewatered sludge becomes promising to some small-scale wastewater treatment plants (WWTPs) especially in the developing countries such as China [8,9]. Although high solid anaerobic digestion of dewatered sludge without pretreatment (feeding TS, 20%) was feasible in the laboratory-scale reactors [8], the rheological behavior of sludge, which is crucial for the design of anaerobic digestion plants, could not be reflected from laboratory-scale experiments because of the scale limitation. The Cambi thermal hydrolysis process (THP) has been applied to several full-scale projects (165–180 °C, 10–30 min, TS = 15–20%) [14], in which the sludge with 12% TS after THP can be handled in the same way as raw sludge with 5–6% TS.

At present, there are still lack of systematical evaluations on the solubilization of organics (soluble proteins, carbohydrates, and volatile fatty acids) and variations in viscosity of high solid sludge after different operational conditions for THP. In this study, the difference of those soluble substances after THP were compared, and further the biogas production of those pretreated sludge in mesophilic batch experiments after THP were checked respectively, by which the appropriate operational conditions for THP and their mechanisms of the acceleration of biogas production were elucidated as the reference for practical applications.

2. Materials and methods

2.1. Sludge characteristics

The dewatered/high solid sludge samples were obtained from a municipal WWTP in Shanghai, China. The samples were stored at 4 °C in laboratory before the experiments. The main characteristics of the sludge used in the experiments are listed in Table 1.

2.2. Pretreatment conditions

The effects of thermal pretreatment depend on treatment temperature and time. In this study, low-temperature thermal hydrolysis process (LTHP: 60 °C, 70 °C, 80 °C, and 90 °C) with different duration time (1 h, 2 h, 4 h, 8 h, 12 h, 24 h, 36 h, 48 h, 60 h, and 72 h) and high-temperature thermal hydrolysis process (HTHP: 120 °C, 140 °C, 160 °C, and 180 °C) with different duration time (15 min, 30 min, 45 min, 60 min, 75 min, 90 min, 105 min, 120 min, 150 min, and 180 min) were carried out to study the comprehensive effects.

In the LTHP, four beakers, each containing 500 g of sludge, were heated in water baths at the different controlled temperatures of 60, 70, 80, and 90 °C for 3 days, respectively. The beakers were covered with lids to prevent water evaporation and magnetically stirred to ensure temperature homogeneity. Around 10 g samples each were taken from the beakers for the measurements at the determined time.

In the HTHP, corresponding to the sampling time, each group contained 10 sealed 100 ml Teflon jars with 70 g sludge (30% safety volume guarantee). The different groups of jars were heated in an oven at the different temperatures (120, 140, 160, and 180 °C, respectively) and taken out one by one at different sampling time. A thermocouple attached to the surface of the jar and a thermodetector were used to measure the surface temperature of the jars, which was approximately as same as the temperature of the sludge inside the jar.

The raw and pretreated sludge samples were analyzed for TS, VS, viscosity, SCOD, VFAs, proteins, carbohydrates, NH_4^+-N, and pH.

2.3. Biodegradability batch tests

The waste activated sludge (WAS) was thermally treated at different temperatures for 24 h (low temperature) or 3 h (high temperature) before mixing with the seed sludge. The seed sludge was pre-incubated to remove its residual biodegradable organics. The pre-incubation was carried out at 35 °C in a water bath for 2 days. The inoculum-to-substrate ratio was set to 2:1 VS (a mixture of 300 g seed sludge and 45 g pretreated WAS). The methane production from only the inoculum was measured, which was subtracted from the total methane production. The main equipment for BMP test was a 500 mL flask incubated at 37 °C in a shaking water bath with 120 rpm. The headspace of each flask was flushed with nitrogen gas for 1 min and immediately sealed using butyl

Table 1
Characteristics of the dewatered sludge.

Parameter	Value
TS (%, w/w)	16.7 ± 0.5
VS/TS	70.5 ± 0.1
TCOD (g/L)	166.0 ± 2.3
SCOD (g/L)	7.55 ± 0.5
Dissolved VFAs (g/L)	1.45 ± 0.1
Dissolved NH_4^+-N (g/L)	1.10 ± 0.1

TS, total solids; VS, volatile solids; TCOD, total chemical oxygen demand; SCOD, soluble chemical oxygen demand; VFAs, volatile fatty acids.

rubber septa. The produced gas was collected in a gas-tight aluminium foil bag during the process, and the volume of the gas was monitored as well. All the experiments were conducted in duplicates. The VFAs, pH, TS, VS, and SCOD values were determined at the end of each batch experiment.

2.4. Analytical methods

The TS and VS/TS contents of the sludge were determined according to the American Public Health Association standard methods [2]. The viscosity, pH, TS, and VS were determined directly. The sludge samples were diluted with ultrapure water to properly measure the other chemical indexes. COD was determined using a Hach DR2000 colorimeter equipped with a Hach COD reactor. For the measurements of SCOD, soluble proteins and carbohydrates, VFAs, and NH_4^+-N, the diluted sludge samples were centrifuged at 10,000*g* for 15 min and filtered through a 0.45 μm microfiber filter prior to the analysis. The soluble protein and carbohydrate concentrations were determined using the Lowry method and Anthrone method, respectively [5].

The viscosity of the raw and pretreated sludge samples were determined at 25 °C using a rotational viscometer (Brookfield DV-I prime, USA). Four different T-bar spindles (C, D, E, and F) were used in the entire study. The torque percentage was adjusted to the range 10–100% by the correct coordination of the rotational speed of the spindle.

The methane content of the biogas was measured using a gas chromatograph (GC, Agilent Technologies, 6890N, CA, USA) equipped with a thermal conductivity detector, Hayseq Q mesh, and Molsieve 5A column.

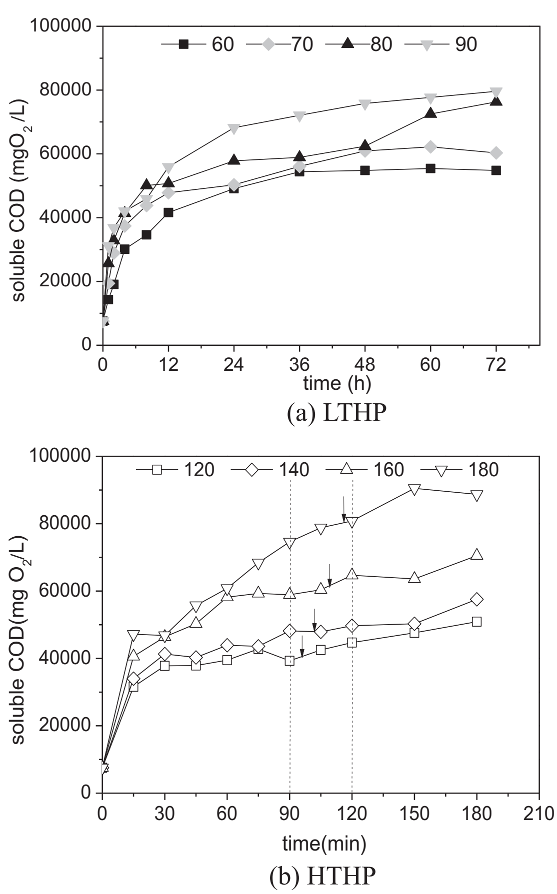

Fig. 1. Concentrations of soluble COD (mgO_2/L) for different treatment temperatures and durations. (a) LTHP, low-temperature thermal hydrolysis process; (b) HTHP, high-temperature thermal hydrolysis process.

To analyze the VFAs, the filtrate was acidified with formic acid to adjust the pH to ~2.0 and mixed on a vortex before the VFAs were analyzed using a GC (Shimadzu GC-2010, Plus column, 30 m × 0.25 mm ID) equipped with a flame ionization detector (FID).

The degree of solubilization of the organics was calculated using the following equation:

$$\text{Soluble COD}(\%) = \frac{\text{Soluble COD g/L}}{\text{Total COD g/L}} \times 100$$

3. Results and discussion

3.1. Effects of pretreatment temperature and time on sludge solubilization

3.1.1. SCOD, proteins, and carbohydrates

Particulate polymeric compounds and microbial cells are disrupted by thermal pretreatment, thus eluting the organics from the cells. Fig. 1(a) and (b) show the increase in the organic content with temperature and time. The particulate insoluble polymers were transformed into soluble monomers and then transferred into the liquid phase, thus increasing the SCOD. The solubilization of organics increased with pretreatment temperature. Thus, temperature had a positive effect on the hydrolysis of organics.

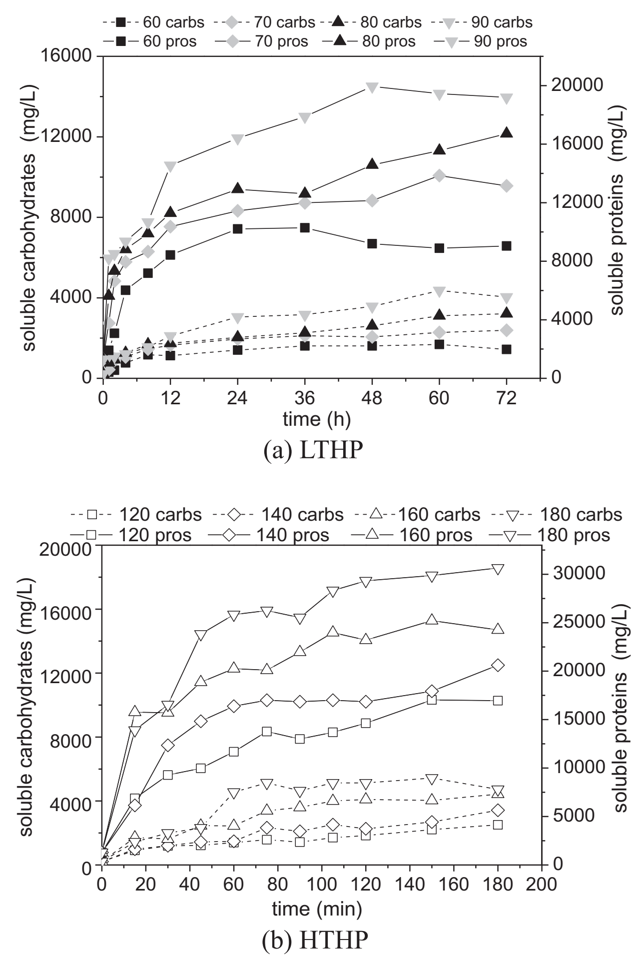

Fig. 2. Concentrations of soluble carbohydrate (mg/L) and soluble proteins (mg/L) for different treatment temperatures and durations. (a) LTHP, low-temperature thermal hydrolysis process; (b) HTHP, high-temperature thermal hydrolysis process.

The concentrations of SCOD rose dramatically with LTHP time in the first 24 h and experienced a slight rise over the next 48 h (Fig. 1(a)). The SCOD of the pretreated sludge after 24 h accounted for 89.6%, 83.4%, 75.8%, and 85.7% of the total SCOD eluted after 72 h at 60 °C, 70 °C, 80 °C, and 90 °C respectively. Thus, from economical perspective, the LTHP treatment time could be set for 24 h. After 24 h treatment, the solubilization of COD increased from 4.5% (raw sludge) to 29.6%, 30.3%, 34.8%, and 41.1% at 60 °C, 70 °C, 80 °C, and 90 °C respectively. The growth of SCOD at 70 °C was not obvious as also reported by Appels et al. [4], which was probably because of the insufficient pretreatment time (60 min) in the study.

In contrast, the HTHP needed 90–120 min (Fig. 1(b)) to reach the set temperature in this study (for specific time, see the black arrow). Since then, there was not significant increase in SCOD. This result is consistent with the literatures, in which the reported SCOD was stable after 10 min of HTHP [7,14]. Overall, 180 min of HTHP (including heating time) was sufficient for the elution of COD. Furthermore, the solubilization of COD increased from 4.5% to 30.7%, 34.7%, 42.5%, and 53.4% at 120 °C, 140 °C, 160 °C, and 180 °C, respectively.

After 24 h LTHP and 180 min HTHP (including the heating time), both the SCOD values were around 50,000–70,000 mg/L, except for that at 180 °C. The drawbacks of low temperature can be offset to a certain degree by increasing the duration time. Compared to HTHP, the effect of LTHP is relatively more dependent on the duration time at the defined temperature.

Carbohydrates and proteins solubilized with thermal pretreatment. Fig. 2(a) and (b) show the changes of the concentrations of proteins and carbohydrates in the soluble phase. After the LTHP for 24 h, the concentrations of soluble proteins at 60 °C, 70 °C, 80 °C, and 90 °C were 6.9, 7.8, 9.0, and 11.7 times of that of the raw sludge, and soluble carbohydrates were 5.7, 8.3, 8.7, and 13.5 times of that of the raw sludge, respectively. Interestingly, the lower the pretreatment temperature, the easier the concentrations of soluble proteins and carbohydrates become stable.

The HTHP exhibited a similar trend to the LTHP, higher temperature and longer heating time resulted in the more soluble substances. The concentrations of carbohydrates and proteins, following the trend of SCOD, became stable after reaching the set temperature.

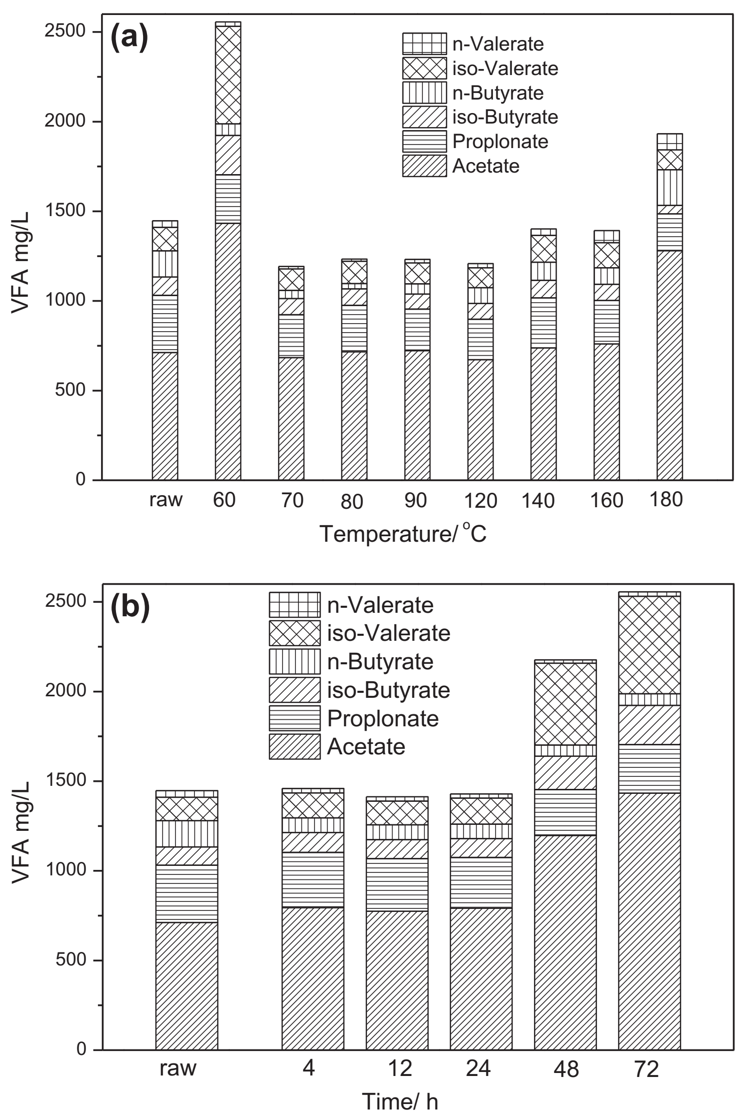

Fig. 3. (a) Concentrations of total volatile fatty acids (VFAs, mg/L) for different treatment temperatures and durations. (b) Concentrations of total volatile fatty acids (VFAs, mg/L) for different pretreatment time at 60 °C.

3.1.2. VFAs and ammonium

Fig. 3(a) shows the concentrations of VFAs in the samples after 72 h LTHP or 180 min HTHP. The concentrations of VFAs remained stable at 70–90 °C during 72 h pretreatment. When treated at 60 °C (Fig 3(b)), the concentrations of VFAs remained stable during the first 24 h, but rose dramatically in the next 2 days. The results were mainly attributed to the increase of acetate and isovalerate, which were nearly double and 4.2-fold after 72-hour pretreatment. In the HTHP, the concentration of VFAs at 180 °C exhibited a relatively high increase compared to that at 120–160 °C, probably because the amino acids decomposed to VFAs above 160 °C [17].

Fig. 4(a) and (b) show the variations in the NH_4^+-N concentration during the LTHP and HTHP. The NH_4^+-N concentration at 60 °C showed a similar trend to the VFAs. The NH_4^+-N concentrations increased slowly during the first 24 h and then grew rapidly over the next 48 h. In contrast, the NH_4^+-N concentrations kept slow increase at 70–90 °C during all the 48 h. In the HTHP, the NH_4^+-N concentrations at 160–180 °C exhibited a relatively high increase compared to that at 120–140 °C. This is similar to that reported by Wilson et al. [17] who found that NH_4^+-N was rapidly released above 170 °C.

The results above show that THP at 70–140 °C only improved the hydrolysis of particulate proteins to soluble proteins (Fig. 2), but contributed little to the subsequent acidogenesis step. The proteins were mostly solubilized instead of being degraded. In contrast, the longer pretreatments at 60 °C may have favored the activities of thermophilic or hyper-thermophilic bacteria, thus promoting enzymatic hydrolysis and affording a pre-digestion step. Unlike the biological effects at 60 °C, the increase of VFAs and NH_4^+-N at 180 °C can be attributed more to a physicochemical effect. Therefore, VFAs and NH_4^+-N can be generated not only by biological reactions, but also by physical reactions.

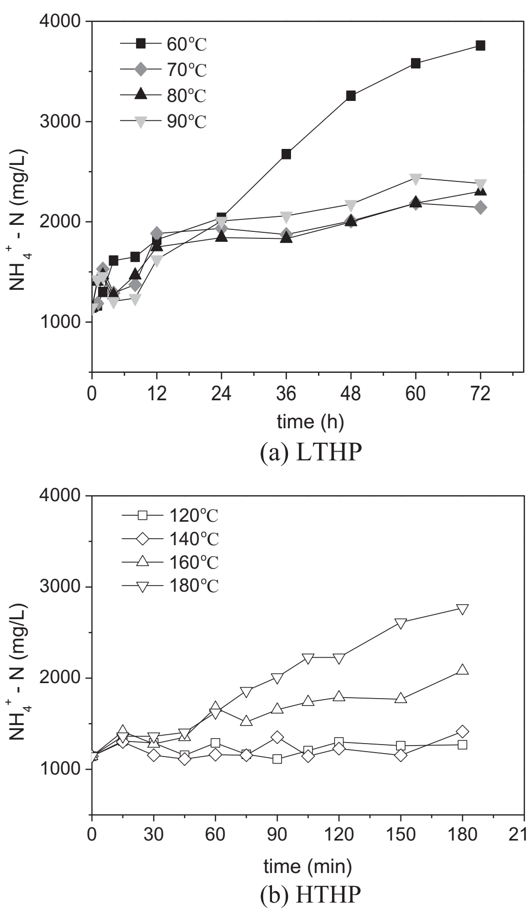

Fig. 4. Concentrations of ammonia nitrogen (NH_4^+-N, mg/L) for different treatment temperatures and durations. (a) LTHP; (b) HTHP.

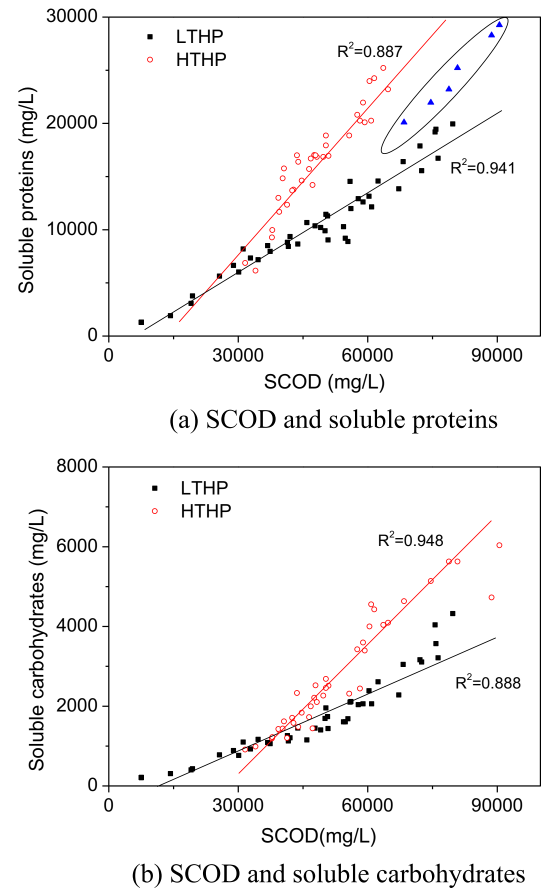

Fig. 5. Correlation between (a) soluble COD and soluble proteins, (b) soluble COD and soluble carbohydrates.

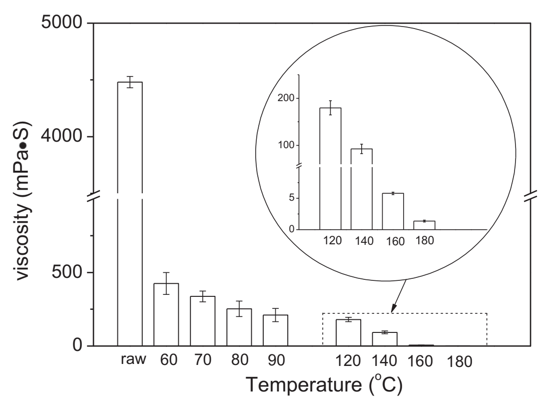

Fig. 6. Effect of pretreatment temperature and time on viscosity.

3.2. Correlation between SCOD and soluble proteins/carbohydrates

Almost all studies on thermal hydrolysis have focused mainly on sludge hydrolysis at various pretreatment temperatures and time. Few studies have been conducted on the correlations between the eluted SCOD and soluble proteins (Spr)/soluble carbohydrates (Sch). Fig. 5(a) and (b) show the relationships between SCOD and Spr, and SCOD and Sch, respectively, during the LTHP

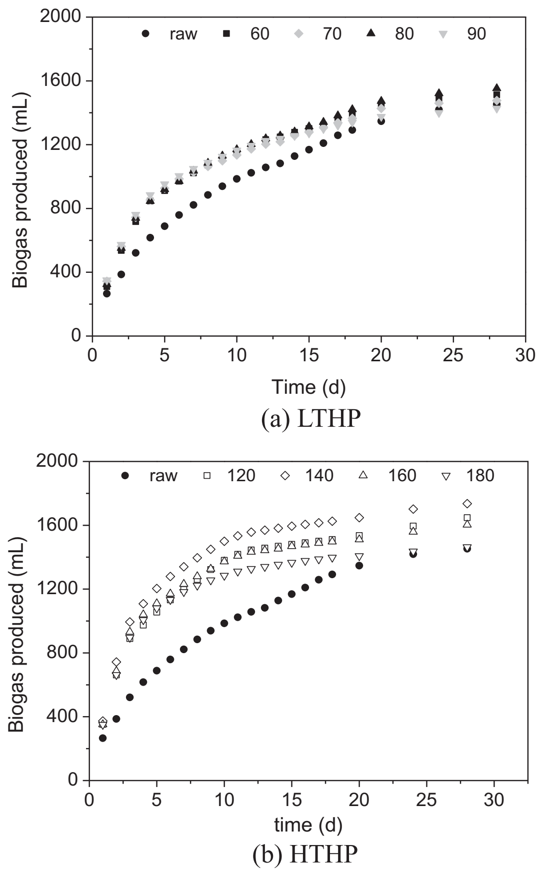

Fig. 7. Results of biogas production after 28 d for (a) LTHP and (b) HTHP.

and HTHP. The SCOD and Spr/Sch exhibited good linear relationships ($R^2 \approx 0.9$, $p < 0.01$), indicating that the particulate proteins, carbohydrates, and other materials contributing to the SCOD were solubilized at the same rate during the LTHP and HTHP. The drastic heating accelerated cell disruption, resulting in abundant soluble proteins and carbohydrates. The results show that the slopes of the lines increased from 0.239 to 0.494 for Spr, and from 0.048 to 0.096 for Sch. The temperature of about 100 °C may be a turning point for the ratios of Spr/SCOD and Sch/SCOD.

However, there were certain points that did not lie on either line (Fig. 5(a)). These phenomena were all happened at 180 °C. At the high temperature, the proteins might have hydrolyzed to individual amino acids that did not react positively in the Lowry assay [17].

3.3. Viscosity

Visually, the sludge was transformed into a more fluid sludge mass after the thermal hydrolysis. The viscosity of sludge exhibited positive changes. Viscosity was selected to characterize the flow state of the sludge.

After the LTHP for 24 h, the viscosity of the sludge decreased from 4480–4530 mPa s to 210–430 mPa s (Fig. 6). Nonetheless, the viscosity of the treated sludge were in the same order of magnitude. However, after the HTHP for 3 h (including heating time), the viscosity significantly reduced to 180, 90, 5.8, and 1.4 mPa s at 120 °C, 140 °C, 160 °C, and 180 °C, respectively. Therefore, the HTHP temperature significantly affected the viscosity of the sludge.

The heat and mass transfer of the sludge can be improved by the decrease in viscosity; thus, energy can be saved during mixing, pumping, and heating [18]. This can balance to some extent the high energy consumed in thermal hydrolysis, particularly for HTHP.

3.4. Biodegradability batch tests

The biodegradability batch experiments were conducted under mesophilic conditions by anaerobic batch experiments using the raw, 24 h LTHP, and 3 h HTHP samples. The results of the experiments are shown in Fig. 7(a) and (b). On day 15, the accumulated gas volumes were 11.3%, 9.3%, 12.3%, and 8.8% for the LTHP samples and 26.6%, 36.4%, 25.6%, and 16.8% for the HTHP samples, higher than that of the untreated sample. In the first two weeks, both the LTHP and HTHP significantly affected anaerobic digestion.

After 28-day digestion, there were not obvious changes in entire gas production between raw and LTHP sludge, indicating that the LTHP improved the early degradation steps of anaerobic digestion (hydrolysis), but slightly affected the latter steps (acetogenesis and methanogenesis). For the HTHP, the total gas volume increased to ~10.6%, 16.5%, and 6.3%, respectively, from 120 °C to 160 °C; conversely, the volume of gas slightly decreased at 180 °C. The volume of gas positively correlated with the reduced VS (Table 2). The

Table 2
Performance of anaerobic digestion system after different pretreatment process.

	Substrate								
	untreated	60 °C	70 °C	80 °C	90 °C	120 °C	140 °C	160 °C	180 °C
Feed composition									
TS (%)	16.73 ± 0.54	16.63 ± 0.11	17.18 ± 0.28	16.82 ± 0.42	16.94 ± 0.22	16.74 ± 0.02	16.89 ± 0.28	16.86 ± 0.18	16.98 ± 0.20
VS/TS (%)	70.48 ± 0.01	71.07 ± 0.26	71.47 ± 0.08	71.29 ± 0.25	71.10 ± 0.21	71.12 ± 0.20	71.11 ± 0.12	71.07 ± 0.17	71.10 ± 0.08
I/S ratio (vs/vs)	2/1	2/1	2/1	2/1	2/1	2/1	2/1	2/1	2/1
pH	7.74	7.72	7.70	7.72	7.76	7.93	7.77	7.82	7.9
Effluent									
TS (%)	9.63 ± 0.2	9.49 ± 0.35	10.03 ± 0.03	9.86 ± 0.06	10.14 ± 0.03	8.64 ± 0.06	8.76 ± 0.1	8.32 ± 0.05	9.83 ± 0.02
VS (%)	33.08 ± 0.03	33.48 ± 0.41	32.02 ± 0.63	31.82 ± 0.46	32.17 ± 0.29	35.26 ± 0.70	33.79 ± 0.08	36.89 ± 0.24	32.80 ± 0.48
VFA (mg/L)	32.9	31.8	30.8	29.5	29.1	33.8	20.2	29.2	15.2
NH_4^+-N (mg/L)	2352	2514	2448	2621	2615	2465	2122	2449	2309
SCOD (mg/L)	1920	2060	2120	2180	2500	2606	3240	3752	6124
VS removal (%)	31.5	31.7	30.9	32.5	29.9	34.5	36.3	34.0	30.7
Biogas characteristics									
Biogas production (L/g VS removed)	0.96	1.01	0.99	1.04	0.97	0.98	0.94	0.98	1.07
Total biogas volume (mL)	1506	1514	1477	1553	1427	1648	1735	1624	1464

TS, total solids; VS, volatile solids; I/S, inoculum-to-substrate ratio; SCOD, soluble chemical oxygen demand; VFAs, volatile fatty acids.

methane concentration in the produced biogas was not significantly affected by the treatment and remained between 65% and 70%.

From Table 2, it was clear that after the digestion, the concentration of VFAs decreased to a low level because of the breakdown during the anaerobic digestion. Unlike the close values of NH_4^+-N concentration at different temperatures, the SCOD showed a higher value at high pretreatment temperatures. In particular, at 180 °C, the SCOD sharply increased. The increase of the SCOD, with poor degradability, and decrease of the gas production indicate that some soluble but non-biodegradable materials were produced at high temperatures. This is probably because of the production of melanoidins, as reported by many studies.

For the raw and LTHP sludge, 18–20 days were needed to reach the level of 90% of the entire gas production. The days required decreased to 15–16, 12–13, and 13–14 days at 120 °C, 140 °C, and 160 °C, respectively. The HTHP at 140–160 °C afforded the best results in terms of gas production. Although the entire gas volume increased by only 6–16%, the SRT could be reduced from 18–20 d to 12–14 d at 140–160 °C. Thus, the effect of thermal hydrolysis is still obvious.

4. Conclusion

Both the LTHP and HTHP positively affected the solubilization of organics in sludge. Treatments with higher temperature and longer duration were beneficial to the solubilization of proteins and carbohydrates, hence accelerated the production of methane in the following high-solid anaerobic digestion.

The fluidization of dewatered sludge was improved after thermal hydrolysis, particularly after the HTHP, which should be further explored in the pilot and large-scale experiments.

In the biodegradability batch experiments, the LTHP did not improve the total gas volume, while the HTHP did. By considering the days needed to obtain 90% of the entire gas volume as the standard, the SRT could be reduced from 18–20 d for the untreated sludge to 12–14 d especially after the thermal hydrolysis under 140–160 °C.

Acknowledgement

This work was financially supported by the key projects of National Water Pollution Control and Management of China (2013ZX07315-001).

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