Effect of steam explosion on physicochemical properties of waste activated sludge and the performance of anaerobic digestion
Yan Zhang, Peng Han, He Liu, Lihui Zhang, Hongbo Liu and Bo Fu

ABSTRACT
The effect of steam explosion on physicochemical properties of sludge and the performance of anaerobic digestion (AD) was investigated. The steam explosion was conducted under different combinations of temperature and time, ranging 151–198 °C and 4–12 min respectively. The capillary suction time (CST) and viscosity of the sludge was increased with particle size decreased by improved hydrolysis temperature and prolonged hydrolysis time. The best sludge solubilization achieved was 41.3% under pretreatment condition of 198 °C and 4 min. Biogas production was enhanced with the improved sludge solubilization, and a linear correlation was found between biogas production and the severity factor (logR0) of steam explosion. However, the biogas productivity was reduced when the logR0 was increased from 3.79 to 3.96, probably owing to the generation of refractory organics during the high severity pretreatment. The temperature of 198 °C and the time of 8 min were the recommended operation parameters of steam explosion pretreatment for sludge AD, which could improve biogas production by 99.7 mL/g VSfed. The pH and NH4+-N during sludge AD was increased by steam explosion pretreatment; however, no inhibition on biogas production was observed.

Key words | anaerobic digestion, biogas production, pretreatment, steam explosion, waste activated sludge

NOMENCLATURE
AD Anaerobic digestion
BC Blank control
COD Chemical oxygen demand
CST Capillary suction time
D50 Median diameter
EC Experimental control
EPS Extracellular polymeric substances
FAN Free ammonia nitrogen
LF Logistic function
MG Modified Gompertz
SCOD Soluble chemical oxygen demand
SR Solubilization ratio
TF Transference function
TP Total phosphorus
TS Total solids
UASB Up-flow anaerobic sludge bed
VFAs Volatile fatty acids
VS Volatile solids
VSfed Volatile solids fed for anaerobic digestion before steam explosion
WAS Waste activated sludge
WWTP Wastewater treatment plant

INTRODUCTION
Waste activated sludge (WAS) is generated as a byproduct of biological wastewater treatment, and is characterized by high concentration of organic matters (Neumann et al. 2016). Owing to its characteristics and the considerable productivity, disposal of WAS is a critical issue accompanying wastewater treatment, but also provides an opportunity for energy and resources recovery. Anaerobic digestion (AD) could effectively achieve mass reduction, pathogen reduction, as well as energy recovery, and is one of the
most widely used sludge treatment technologies. A combustible biogas composed principally of CH₄ and CO₂ is produced by AD from organic substrate in the WAS via hydrolysis, acidogenesis, acetogenesis and methanogenesis. However, intracellular organic materials of the sludge are segregated by microbial cell walls, which are hard to degrade during hydrolysis, and often cause lowering of overall sludge digestion efficiency (Lee et al. 2017). Therefore, hydrolysis is regarded as the limiting step of AD and the limiting factor influencing biogas production (Gianico et al. 2013). In order to speed up the reaction rate and enhance reaction efficiency of digestion, various pre-treatment technologies have been studied to overcome the drawback of hydrolysis, including physical technologies like thermal treatment (Bougrier et al. 2007), chemical technologies like alkaline (Lin et al. 2009) and acid (Devlin et al. 2011) treatment, Fenton (Pilli et al. 2016), and biological technologies like enzyme treatment (Yin et al. 2016). Among these pre-treatment technologies, thermal hydrolysis was the first applied since late 1970, and is recognized as one of the most common and successful pretreatment methods for its significant enhancement of AD performance (Li et al. 2017). Two main thermal hydrolysis processes are developed based on different applied temperatures: low temperature thermal hydrolysis applying moderate temperatures (normally under 100 °C (Appels et al. 2010)) and high temperature thermal hydrolysis with temperature of 100–210 °C (Neumann et al. 2016). Owing to the relative low applied temperature, it took several hours to solubilize organic matters in sludge by low temperature hydrolysis (Climent et al. 2007), while only 20–60 min was needed to achieve 12–80% chemical oxygen demand (COD) solubilization by high temperature treatment (Neumann et al. 2016). Hydrolysis temperature and hydrolysis time are two crucial variables for thermal hydrolysis. An improved temperature can significantly shorten pretreatment time; however, recalcitrant compounds may generate under temperatures over 170–190 °C (Carrere et al. 2010), which may further inhibit biogas production. Therefore, an optimum range of hydrolysis temperature and hydrolysis time is recommended for sludge pretreatment.

Steam explosion is a variation of high temperature thermal hydrolysis, which consists of the application of high temperature with high pressure steam in a reaction vessel, and a sudden release of the vapor pressure (called flashing). The sudden release of the pressure produces a strong shear effect on the sludge that further enhances the sludge disintegration after high temperature thermal treatment. Therefore, steam explosion could achieve better pretreatment performance than thermal hydrolysis under the same thermal treatment condition. Its enhancement performance for AD of WAS (Dereix et al. 2006) as well as pig manure (Ferreira et al. 2014), olive mill solid waste (Rincon et al. 2016), and rice straw (Zhou et al. 2016) has been reported. However, the effect of the two key operation parameters, hydrolysis temperature and hydrolysis time, of steam explosion on solubilization and AD of WAS was rarely studied (Perez-Elvira et al. 2015; Sapkaite et al. 2017). As steam explosion supplemented a mechanical disintegration step by flashing after thermal treatment, optimum ranges of operation variables for high temperature thermal hydrolysis can not be applied to steam explosion directly. Perez-Elvira et al. (2015) studied the relationship between the severity factor (logR₀) of steam explosion and sludge solubilization, and biochemical methane potential; however, the influence of each of the two key operation variables was not clarified. Sapkaite et al. (2017) investigated the influence of hydrolysis temperature and hydrolysis time on biochemical methane potential; however the impact of steam explosion on methane production rate was not analyzed. Besides, steam explosion influences physicochemical properties, which further influence followed treatment process. For example, particle size may influence the efficiency of sludge dewatering, and viscosity may also influence dewatering efficiency as well as the agitation power during digestion. However, the variation of physicochemical properties of sludge treated with steam explosion has not been investigated yet.

This study conducted sludge pretreatment by steam explosion under 12 operation conditions combined with different hydrolysis temperatures and times (Table 1). The

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<th>Table 1</th>
<th>Operating parameters and severity factor of steam explosion adopted in this study</th>
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effect of steam explosion under different operation conditions on the main physicochemical properties as well as AD performance of WAS were revealed.

**MATERIALS AND METHODS**

**Sludge**

The substrate for steam explosion and digestion, WAS, was taken from a dewatered sludge tank of a local wastewater treatment plant (WWTP), in which the sludge was predewatered by a belt press filter. The seed anaerobic sludge, which provided anaerobic microorganisms for digestion, was anaerobic granular sludge taken from an up-flow anaerobic sludge bed of another local WWTP in Wuxi city, China. The WAS had pH of 7.3, total solids (TS) concentration of 136 g/L, volatile solids (VS)/TS ratio of 53.67%, soluble chemical oxygen demand (SCOD) of 761.5 mg/L, soluble protein of 71.6 mg/L, soluble polysaccharide of 88.4 mg/L, ammonium nitrogen ($NH_4^+$-N) of 52.8 mg/L, and total phosphorus (TP) of 8.2 mg/L. The seed anaerobic sludge had pH of 7.5 mg/L, TS of 110 mg/L, VS/TS ratio of 68.52%, and $NH_4^+$-N of 526.6 mg/L.

**Steam explosion device and operation parameters**

A steam-explosion machine (QBS-80, Zhengdao, China) with heating power of 8 kW, maximum vapor pressure of 3.2 MPa, cylinder capacity of 400 mL, was used in this study. A 200 g WAS was treated in each steam explosion batch. Total 12 sets of steam explosion parameters, which covered four different hydrolysis temperatures and three different hydrolysis times, were applied. The detailed steam explosion parameters are shown in Table 1. The severity factor, $logR_0$, of thermal hydrolysis was calculated by Equation (1) (Ferreira et al. 2014).

$$logR_0 = log \left( t \times \exp \left( \frac{T - 100}{14.75} \right) \right)$$  \hspace{1cm} (1)

T is the pretreatment temperature, °C; t is the pretreatment time, min.

**Sludge AD**

AD was carried out in 1 L glass conical flasks containing 400 mL steam explosion pretreated WAS as substrate and 80 mL seed anaerobic sludge providing anaerobic microorganisms. After sludge was added, the headspace of the flask was filled with N$_2$ gas for 5 min to eliminate O$_2$. Then, the flask was sealed with a rubber stopper, in which a glass tube was inserted to collect biogas. 400 mL un-pretreated WAS digested with 80 mL seed anaerobic sludge was conducted as the experimental control (EC). Meanwhile, 80 mL seed anaerobic sludge digested with no WAS feeding was conducted as the blank control (BC). All the digestion experiments were implemented in an oscillating water bath under 35 °C for 30 days. The pH, SCOD, and $NH_4^+$-N in the digestion liquid were determined daily.

**Analytical methods**

Conventional indices, including TV, VS, $NH_4^+$-N, TP, SCOD, and pH were analyzed according to the standard methods. Particle size distribution was measured by a laser particle size distribution instrument (BT-2003, Bettersize, China). Capillary suction time (CST) was tested by a Type 304M CST analyzer. Viscosity was determined by a rheometer (LVDV-3T, Brookfield, MA). The soluble phase was obtained by centrifugation at 10,000 rpm for 5 min, and filtrated with a 0.45 μm membrane. The concentrations of polysaccharide and protein were measured by the phenol-sulfuric acid method (Dubois et al. 1956) and the Coomassie brilliant blue staining method (Bradford 1976), respectively.

The solubilization of the WAS treated with steam explosion was assessed with solubilization ratio (SR), which was calculated by Equation (2).

$$SR = \frac{VS_0 - VS_t}{VS_0} \times 100\%$$  \hspace{1cm} (2)

$VS_0$ is the initial VS of the WAS before steam explosion pretreatment; $VS_t$ is the VS of the WAS after steam explosion pretreatment.

The volume of produced biogas was measured by displacement of saturated aqueous NaCl in a graduated measuring cylinder (Abouelenien et al. 2009). The volume of cumulative biogas from sludge digestion systems was amended with that from BC, which was the biogas produced by endogenous respiration of the seed anaerobic sludge. The specific biogas production rate was obtained by normalizing the volume of cumulative biogas with initial VS fed for AD before steam explosion ($VS_{fed}$). Therefore, the effect of steam explosion operation condition on the performance of sludge AD could be investigated by comparing specific
biogas production rate. Methane content in biogas was determined by a GC (GC9790II, Fuli, China).

**Modelling**

The biogas production curves were fitted to three models in order to obtain the kinetic parameters. The three considered biogas production models were modified Gompertz (MG) (Nopharatana et al. 2007), transfer function (TF) (Donoso-Bravo et al. 2011), and logistic function (LF) (Donoso-Bravo et al. 2010), shown as Equations (3)–(5), respectively.

\[
B = P \times \exp \left\{ -\exp \left[ \frac{R_m \times e}{P} (\lambda - t) + 1 \right]\right\} \quad (3)
\]

\[
B = P \times \left[ 1 - \exp \left( -\frac{R_m (t - \lambda)}{P} \right) \right] \quad (4)
\]

\[
B = \frac{P}{1 + \exp \left( \frac{4R_m(\lambda - t)}{P} + 2 \right)} \quad (5)
\]

In the models, \( B \) is the cumulative biogas production (mL), \( P \) is biogas production potential (mL), \( R_m \) is the maximum biogas production rate (mL/d), \( \lambda \) is the duration of the lag phase (d), and \( t \) is the duration of the digestion at which cumulative biogas production, \( B \), is calculated (d). Adjusted \( R^2 \) (adj. \( R^2 \)) was calculated to evaluate the accuracy of each model. The \( R_m \) obtained from the most accurate model was used to compare the maximum biogas production rate of the digestion of pretreated WAS under different steam explosion condition.

**RESULTS AND DISCUSSION**

**Changes in physicochemical properties of the sludge**

Particle size distribution, CST, and viscosity of the sludge before and after steam explosion were measured. As shown in Figure 1(a), sludge particle size was decreased by steam explosion pretreatment. The decrease was generally enlarged with hydrolysis temperature improvement and hydrolysis time extension. The median diameter (D50) was decreased from 61.56 μm in raw sludge to 28.9 μm in that treated under 198°C for 8 min. The decreased particle size was also observed during hydrothermal (Park et al. 2017) and other pretreatment (Zhang et al. 2016), which was probably owing to flocculation disintegration caused by solubilization of extracellular polymeric substances (EPS) and lysed cells. However, when the hydrolysis time was extended to 12 min under 198°C, the particle size of the sludge rebounded to D50 of 58.91 μm, which was very close to that of the raw sludge.

CST and viscosity were gradually increased with the improvement of steam explosion severity, e.g. increased hydrolysis temperature and prolonged hydrolysis time (shown in Figure 1(b)). As EPS often positively influence CST (Neyens et al. 2004) and viscosity (Li & Yang 2007), the significantly increased CST and viscosity, from 17.1 s and 201.9 cP in the raw sludge to 690.0 s and 520.8 cP in the sludge treated under 198°C for 8 min, indicated a high possibility of enhanced extracellular organic matter in the sludge with steam explosion pretreatment. The increased CST indicated that the dewaterability of the sludge was deteriorated, which occurs frequently after pretreatment due to the destruction of sludge flocs and the release of EPS (Zhu et al. 2015; Liu et al. 2017). The deterioration of sludge dewaterability caused by steam explosion was lower than that caused by thermal alkaline hydrolysis, which could increase CST of the sludge from 40–50 s to about 2,500 s under 90°C, pH 12.0 of 3 h (Liu et al. 2017). The less deterioration of sludge dewaterability could be one of the advantages of steam explosion for sludge pretreatment.

The CST and viscosity of the sludge treated under 198°C 12 min were 707.2 s and 505.2 cP respectively, similar to that of the sludge treated under 198°C for 8 min and much higher than that of the raw sludge. This indicated that a large amount of intracellular organic matters may also released under 198°C for 12 min. The increased sludge particle size when hydrolysis time is prolonged from 8 min to 12 min under 198°C was probably attributed to the re-aggregation of the sludge after disintegration and solubilization under high pretreatment severity.

**Solubilization of organic matters in steam exploded sludge**

The SCOD, soluble polysaccharide, soluble protein, NH₃-N, and TP concentration in the liquid phase as well as the SR was determined to evaluate the solubilization of the sludge treated by steam explosion. As shown in Figure 2(a), the SR of the sludge treated by steam explosion ranged from 23.9–41.3%, indicated a good solubilization of steam explosion treated sludge. The highest SR appeared in the sludge treated with 198°C for 12 min, while the lowest SR showed in the sludge treated with 151°C for 4 min. The SR seems to be improved with the increasing of hydrolysis temperature and
extending of hydrolysis time. Consistently, the SCOD in the sludge increased from 761 mg/L to 6,396–9,718 mg/L with the pretreatment of steam explosion, and the soluble polysaccharide and protein were improved from 88 mg/L and 71 mg/L to 1,457 mg/L and 866 mg/L, respectively. Both polysaccharide and protein showed a good linear correlation with SCOD, with Pearson r of 0.9553 and 0.9379 respectively (shown in Figure S1, available with the online version of this paper). However, polysaccharide increased faster than protein with SCOD increasing, which indicated that the intracellular polysaccharide released more and faster than protein during steam explosion pretreatment. A high proportion of protein in the substrate will result in a high concentration of ammonia, which may substantially influence AD performance (Chen et al. 2008). The more released polysaccharide with steam explosion pretreatment could increase the C/N ratio of the available substrate in the sludge, and benefit biogas production.

NH₄⁺-N and TP in the liquid phase were also increased significantly after steam explosion, which ranged 425–1,032 mg/L and 41–98 mg/L, respectively (Figure 2(b)). There was a substantial increase of NH₄⁺-N concentration when the hydrolysis temperature increased from 164 °C to 179 °C, which was consistent with the finding by Wilson.
& Novak (2009), e.g. the release of NH$_4^+$-N was significantly improved when hydrolysis temperature was over 170°C. The TP concentration was gradually increased with hydrolysis time extension under each treatment temperature. The variation of NH$_4^+$-N concentration was consistent with TP under temperatures of 151°C, 164°C, and 179°C. However, NH$_4^+$-N concentration decreased with hydrolysis time prolonged from 8 min to 12 min under 198°C. The decreased soluble NH$_4^+$-N concentration in steam explosion treated sludge was also found when hydrolysis temperature increased from 180°C to 260°C (Dereix et al. 2006); however the possible reason was not discussed. As melanoidins could probably generate when the hydrolysis temperature was relatively high of 180°C via Maillard reaction (Wilson & Novak 2009), which polymerized amino acids and saccharides, the decreased NH$_4^+$-N could probably be attributed to melanoidins generation.

According to the SR of the WAS and concentration improvement of the SCOD, soluble polysaccharide, soluble polysaccharide, NH$_4^+$-N, and TP, the enhancement of sludge solubilization by steam explosion treatment was revealed. Wang et al. (2009) also found the effective release
of polysaccharide and other biodegradable compounds from steam explosion treated straw, demonstrated the good pre-treatment performance of steam explosion.

**The relationship of steam explosion and organics solubilization**

The logR₀ is an effective factor to represent the severity of steam explosion, which directly influences sludge pretreatment performance. The relationships between logR₀ and SR, and SCOD were evaluated to investigate the effect of steam explosion severity on the sludge solubilization. As shown in Figure 3, both SR and SCOD showed significant linear correlations with logR₀, indicated that a higher steam explosion severity could result in a better solubilization of sludge. Hydrolysis temperature and hydrolysis time are the two parameters determining steam explosion treatment severity. With one of the two factors kept constant, increase of the other improves the steam explosion severity, thus may enhance the solubilization of the treated sludge. Consequently, the SCOD concentration in the WAS treated with higher hydrolysis temperature and longer hydrolysis time was generally higher (shown in Figure 4). However, under different hydrolysis time, the enhanced magnitude of sludge solubilization by increased hydrolysis temperature was not consistent. As shown in Figure 4(a), when hydrolysis time was relatively short at 4 min, the slope of the SCOD curve from temperature 151 to 164 °C was significantly lower than that in the range of 164–198 °C. It indicated that the specific solubilization enhancement rate by increasing temperature in the range of 151–164 °C was less than that in the range of 164–198 °C. Similarly, when hydrolysis time was 8 min and 12 min, the specific solubilization enhancement rate by increasing temperature in relatively low temperature range (151–179 °C) was slightly lower than in the higher temperature range (179–198 °C). It revealed that increasing hydrolysis temperature in the relatively high temperature range could improve more sludge solubilization, which was probably owing to higher hydrolysis rate under high temperature.

In addition, different enhancement of sludge solubilization by increased hydrolysis time under different hydrolysis temperature was also revealed (shown in Figure 5(b)). The slope of SCOD curve from 8–12 min of 151 °C was higher than that from 4–8 min, indicating a better solubilization enhancement by extending hydrolysis time in the longer hydrolysis time range. However, the slope of the two fragments of SCOD curve of 164 °C was similar; and the curves from

![Figure 3](image1.png) **Figure 3** | The correlation between SR, SCOD and logR₀.

![Figure 4](image2.png) **Figure 4** | Effect of (a) hydrolysis temperature and (b) hydrolysis time on sludge solubilization.
4–8 min of 179 °C and 198 °C were steeper than their corresponding fragments from 8–12 min, which was contrary to that of 151 °C. This phenomenon revealed that when hydrolysis temperature was relatively low at 151 °C, a longer hydrolysis time was recommended for effectively increasing the sludge solubilization; while under a higher hydrolysis temperature of 198 °C, the hydrolysis time could be shorter. Xue et al. (2015) revealed that a longer hydrolysis time (24 h) was needed to effectively hydrolyze sludge by low-temperature thermal hydrolysis (60–90 °C), while only 180 min was required to achieve a good pretreatment performance by high-temperature thermal hydrolysis (120–180 °C). Higher temperature significantly increases the sludge solubilization and organics release. The reported hydrolysis time for high-temperature thermal hydrolysis (20–60 min) was much shorter than that for lower-temperature thermal hydrolysis (up to 7 days) (Neumann et al. 2016). Steam explosion has the advantages of high-temperature thermal hydrolysis, and developed a better means, flashing, for sludge disintegration. Therefore, steam explosion could achieve an excellent hydrolysis performance within several minutes (Zhou et al. 2016), much shorter than that for high-temperature thermal hydrolysis.

**Biogas production from steam explosion pretreated sludge**

The biogas production by AD from steam explosion pretreated sludge was investigated. The cumulative biogas amount is shown in Figure 5. The amount of produced biogas from steam explosion pretreatment sludge (1,651–2,930 mL) was significantly more than that from un-pretreated sludge (537 mL) after 30 days’ digestion.

To further compare the effect of steam explosion operation condition on performance of sludge AD, specific biogas production rate was calculated by normalizing the volume of cumulative biogas with VSfed. The specific biogas production rate was improved from 22.4 mL/g VSfed to 68.8–122.1 mL/g VSfed by steam explosion pretreatment. Moreover, the biogas was produced faster from steam explosion pretreated sludge than the untreated sludge. After pretreatment, biogas generated fastest at the beginning of the digestion, and gradually slowed down as the digestion continued. The proportion of the biogas produced in the first 6 days was 43.5–62.7% in the pretreated sludge digestion system, which was much higher than 27.0% in the EC. The improved biogas amount and the accelerated biogas production demonstrated the excellent pretreatment performance of steam explosion. Consistently, the biogas production was significantly accelerated from pig manure by steam explosion treatment under 170 °C for 30 min, the specific biogas production rate was two times more than that of the un-pretreated control (Ferreira et al. 2014). Besides biogas production, methane content in the biogas was also improved from 18.3% in the EC to around 50% in the pretreated sludge digestion system. The relatively low specific biogas production rate (22.4–122.1 mL/g VSfed) in this study was attributed to the low inoculation rate of seed anaerobic sludge (16.7%). High inoculation
rate of seed anaerobic sludge could significantly increase the biogas production rate of AD (Liu et al. 2009).

The biogas production curves were fitted to three models in order to obtain the kinetic parameters. As shown in Table S1 (available with the online version of this paper), the average adj. R² of model TF fitting for all the biogas production curves was 0.9817, which was higher than that of model MG (average adj. R² = 0.9487) and model LF (average adj. R² = 0.9371). It indicated that model TF was the most accurate one for biogas production modelling in this study. Therefore, Rm calculated by model TF (shown in Table S1) was used to analyze the influence of steam explosion on the maximum biogas production rate. It was found that the Rm significantly increased from 26.3 mL/d in the EC to 225.7–399.1 mL/d in the digestion system of pretreated sludge, indicating the improved maximum biogas production rate along with the specific biogas production rate by steam explosion.

The relationship of steam explosion and biogas production improvement

The effect of steam explosion variables on biogas production improvement was investigated. It is revealed in Figure 5 that cumulative biogas production was improved with hydrolysis time extended under the same hydrolysis temperatures of 151 °C, 164 °C, and 179 °C, but not 198 °C. The relationship between improved biogas production and hydrolysis temperature showed the same phenomenon (shown in Figure 6(a)). Under 151 °C, the improved biogas production was significantly enhanced with extended hydrolysis time. While under 164 °C and 179 °C, the improved biogas production was also enhanced with extended hydrolysis time, though the enhancement from 8 min to 12 min was slight. However, when the hydrolysis time was prolonged from 8 min to 12 min under 198 °C, the improvement of biogas production was reduced, though the solubilization of the sludge was the best (Figure 2(a)). It indicated that high sludge solubilization caused by steam explosion is not bound to a good biogas production if the sludge is pretreated under a high temperature for an overlong time. Consistent with biogas production, the Rm was improved by prolonging hydrolysis time under hydrolysis temperatures of 151 °C, 164 °C, and 179 °C; however, the Rm was significantly decreased when the hydrolysis time was extended from 8 min to 12 min under hydrolysis temperature of 198 °C. From the relationship between hydrolysis temperature and improved biogas production (Figure 6(c)), it can be seen that the enhancement of biogas production with hydrolysis temperature improvement under hydrolysis time of 12 min was less significant than that under hydrolysis times of 4 min and 8 min. Similarly, when hydrolysis temperature increased to 198 °C, the Rm was significantly enhanced with hydrolysis times of 4 min and 8 min; however, it decreased under hydrolysis time of 12 min (shown in Figure 6(d)).

The relationship between improved biogas production, Rm and logR0 was evaluated and is shown in Figure 6(e) and 6(f) respectively. Generally, both biogas production and Rm showed improvement with increased logR0. However, both biogas production and Rm significantly decreased under the highest logR0 in this study (logR0 = 3.96). If the data at logR0 of 3.96 is excluded, biogas production and Rm showed better linear correlations with logR0, with Pearson r of 0.9492 and 0.9171 respectively. Compared to the correlation between sludge solubilization and logR0 (Figure 3), it is revealed that increasing logR0 generally could enhance both sludge solubilization and biogas production. However, when logR0 was 3.96, though a better sludge solubilization was achieved, the biogas production significantly decreased compared to logR0 of 3.79. Similarly, Ferreira et al. (2014) found that the methane productivity and Rm under logR0 of 3.85 was substantially reduced compared to that under logR0 of 3.55, when applying steam explosion to pig manure pretreatment. Perez-Elvira et al. (2015) revealed that the Rm of the sludge pretreated under logR0 of 4.1 and 3.8 was also significantly decreased compared to that under logR0 of 3.1. As shown in Figure S2 (available with the online version of this paper), the residual SCOD in the sludge after digestion was gradually increased from 612 mg/L in the EC to 1,723 mg/L in the sludge treated under 198 °C for 8 min. A linear correlation was revealed between SCOD and logR0 when the logR0 was less than 3.80. However, the residual SCOD substantially increased to 3,245 mg/L under logR0 of 3.96. Consistent with the relatively low concentration of NH4+-N in the steam exploded sludge (Figure 2(b)), the increased SCOD in the sludge after digestion also indicated that more refractory organics like melanoidins were probably generated under high logR0 of 3.96, which resulted in a relatively low biogas production. Therefore, it was inappropriate to extend hydrolysis time as long as 12 min under hydrolysis temperature of 198 °C. A severity of logR0 lower than 3.96 was recommended in steam explosion for sludge pretreatment.

Effect of steam explosion pretreatment on pH and NH4+-N during sludge digestion

The pH and NH4+-N concentration are two important factors which may greatly influence the performance of AD.
If pH and NH₄⁺-N concentration deviate from the optimum range, the activity of anaerobic microorganisms will be inhibited and the digestion may fail. Therefore, the effect of steam explosion on pH and NH₄⁺-N was also investigated.

The variation of pH during sludge AD is shown in Figure S3 (available with the online version of this paper). In EC, pH decreased from 6.79 to 6.71 in the first two days of the digestion. The decrease of pH may be owing to volatile fatty acid (VFA) accumulation in the digestion system. Subsequently, the pH in EC increased to 7.27 on day 5, and fluctuated between 7.12–7.38. In the pretreated sludge digestion systems, the pH directly increased from 6.61–6.92 to 7.18–7.51 in the first two days of digestion, then kept relatively steady in 7.34–7.69. Steam explosion pretreatment accelerated biogas production, avoided pH reduction caused by VFA accumulation at the initial stage of digestion, and increased the final pH to 7.43–7.72, which was well within the optimum pH range for methane production by AD.

**Figure 6** Relationship between improved biogas production and (a) hydrolysis time, (c) hydrolysis temperature, (e) severity factor; and between improved Rₚ and (b) hydrolysis time, (d) hydrolysis temperature, (f) severity factor.
A high NH$_4^+$-N concentration could induce a high concentration of free ammonia nitrogen (FAN), which has been suggested to be the main cause of inhibition since it is freely membrane permeable. The inhibition concentration of FAN could be dozens to hundreds of mg/L in AD systems with different substrates, microorganisms, digestion temperature, etc. (Chen et al. 2008; Kim et al. 2011; Hidaka et al. 2013). During digestion, the initial NH$_4^+$-N concentration in the pre-treated sludge was 570–1,087 mg/L higher than the EC, owing to the enhanced release of intracellular organics by steam explosion. The NH$_4^+$-N concentration in the pretreated sludge digestion system gradually increased in the first 15 days, then kept steady in the following digestion period. As shown in Figure S4, maximum NH$_4^+$-N concentration during digestion was increased by 459–72.25 mg/L in the steam explosion pretreated sludge digestion systems comparing to that in the EC. The FAN during AD was calculated according to Angelidaki & Ahring (1995), and was found at 3.43–72.25 mg/L in the steam explosion pretreated sludge systems higher than that in the un-pretreated ones (0.33–9.66 mg/L). Fortunately, the highest NH$_4^+$-N concentration and FAN concentration in each pretreated sludge digestion system was 938–1,543 mg/L and 37.22–72.25 mg/L, respectively, which was considered as a safe concentration for biogas production (Chen et al. 2008; Duan et al. 2012).

CONCLUSIONS

The influence of key operation parameters of steam explosion on physicochemical properties and AD of WAS was investigated. Steam explosion decreased the particle size and increased the CST and viscosity of WAS. The improvement of sludge solubilization, biogas production, and maximum biogas production rate was obtained under all the operation conditions of steam explosion. Better sludge solubilization was obtained under the largest steam explosion severity of 3.96; however, the highest biogas production was achieved under log$_R$$_0$ of 3.79. The results suggest that 198 °C and 8 min (log$_R$$_0$ = 3.79) are the optimum operation parameters of steam explosion for WAS pretreatment, which could improve biogas production by 99.7 mL/g VS$_{fed}$. The pH and NH$_4^+$-N during sludge AD was increased by steam explosion pretreatment; however, no inhibition on biogas production was observed.

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CONFLICTS OF INTEREST

None.

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