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Influence of NaOH and thermal pretreatment on dewatered activated sludge solubilisation and subsequent anaerobic digestion: Focused on high-solid state

Shuting Zhang [a], Haigang Guo [a], Lianzhu Du [b], Junfeng Liang [b], Xuebin Lu [a], Nan Li [a], Keqiang Zhang [b,*]

[a] School of Environmental Science and Engineering, Tianjin University, Tianjin 300072, China
[b] Agro-Environmental Protection Institute, Ministry of Agriculture, Tianjin 300191, China

HIGHLIGHTS

- Comparative effects of NaOH and thermal pretreatment on dewatered sludge.
- A combination of high-solid pretreatment and high-solid digestion.
- Cumulative methane yield was improved by 71% after 120 °C pretreatment.

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ABSTRACT

In this study, the influence of NaOH and thermal pretreatment of dewatered activated sludge (DAS) on the high-solid solubilisation and anaerobic digestion was separately investigated by monitoring common parameters. The results indicated that COD, proteins and carbohydrates were efficiently solubilised in both NaOH and thermal pretreated DAS samples. For NaOH pretreatment, the concentrations of volatile fatty acids (VFAs) and total ammonium nitrogen (TAN) firstly increased followed by decreasing with NaOH dose increasing. However, they decreased with the severity of thermal pretreatment. During the batch digestion experiments (at 37 °C), for 80 mg NaOH g^{-1} total solid (TS) DAS pretreatment it resulted in a 6.99% decrease in cumulative methane yield (CMY) compared to untreated DAS. While for 80, 100, 120 °C and 20 mg NaOH pretreatment, CMY increased by 15%, 42%, 71% and 35%, respectively, in comparison to untreated DAS.

1. Introduction

Waste activated sludge is the byproduct during wastewater treatment process, and sludge handling accounts for about 50% of the operating cost of many wastewater treatment plants. Especially in China, about 30 million tons of sewage sludge (80% moisture content) are generated annually, and almost 80% of the sludge has no necessary stabilisation (Duan et al., 2012), which results in a big burden to society and environment. It is therefore necessary to stabilise the sludge via promising treatments. Anaerobic digestion is a useful technology to stabilise organic wastes and simultaneously produce renewable energy biogas. It has been widely implemented in kinds of organic wastes, such as agricultural wastes, food wastes and sewage sludge (Dearman and Bentham, 2007; Raheman and Mondal, 2013).

However, the anaerobic digestion is usually carried out at low-solid state (TS < 15%). Compared with low-solid anaerobic digestion, high-solid anaerobic digestion (TS ⩾ 15%) has many advantages, such as smaller reactor volume, less energy input for heating, minimal material handling and so on (Guendouz et al., 2008). In addition, more than 80% of the sewage sludge has already been dewatered before further disposal in China, which makes it favourable to centralised treatment by high-solid anaerobic digestion (Dai et al., 2013), avoiding secondary sludge dehydration by low-solid anaerobic digestion.

Nowadays, high-solid anaerobic digestion of the sludge has been studied more and more in sludge treatment, and it was confirmed to have good operating performances (Duan et al., 2012; Hidaka et al., 2013; Li et al., 2014). However, high-solid anaerobic digestion results in a decrease in methane yield per g volatile solid (VS). To increase the biogas production, lots of researches have been conducted. For example, the addition of scrap iron enhanced high-solids anaerobic digestion of waste activated sludge (Zhang

* Corresponding author. Tel.: +86 22 23616689.
E-mail address: keqiangzhang68@163.com (K. Zhang).

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et al., 2014b). Anaerobic co-digestion of dewatered sludge was also carried out with pig manure, food waste and organic amendments (Zhang et al., 2014a; Dai et al., 2013; Komilis et al., 2011). Nevertheless, anaerobic digestion is a series of reactions involving hydrolysis, acidogenesis and methanogenesis. It is considered that the hydrolysis stage is a rate-limited step in the anaerobic digestion of waste sludge (Batstone et al., 2009). Many pretreatment methods are therefore implemented to improve the solubilisation of the sludge in the hydrolysis process, such as alkaline, thermal, microwave, ultrasound, biological pretreatment (Li et al., 2012; Jang and Ahn, 2013; Gianico et al., 2013; Le et al., 2013; Feng et al., 2015). Recently, NaOH and thermal pretreatment methods have been more and more studied for further enhancing sludge solubilisation and methane yield, and they showed a good pretreatment performance. Li et al. (2012) and Cho et al. (2014) investigated the NaOH pretreatment of the sludge, and discovered that the solubilisation efficiency and methane yield increased significantly. Gianico et al. (2013) concluded anaerobic digestion of thermal pretreated sludge gained significant soluble COD removal and more methane yield. Moreover, the anaerobic digestion of waste activated sludge achieved a 34.8% methane potential increase and a doubled methane production rate by using thermal pretreatment at 175 °C/60 min (Liu et al., 2012). However, their researches was only concentrated on low-solid anaerobic digestion of the sludge.

So far, there has been a lack of study referred to the effect of NaOH and thermal pretreatment on DAS solubilisation and subsequent high-solid anaerobic digestion. The whole assessment has not been carried out for NaOH and thermal pretreatment at high-solid state and was therefore the objective of this research. In current work, the concentrations of COD, carbohydrates, proteins, VFAs and TAN were monitored before and after pretreatment, which was aim at investigating the effects of NaOH and thermal pretreatment on DAS solubilisation. In order to better understand the performance of pretreatment on biogas production, daily methane yield was monitored during anaerobic digestion. COD, carbohydrates, proteins, VFAs and TAN were also determined in pretreated DAS samples after anaerobic digestion.

2. Methods

2.1. Substrates and inoculums

DAS used in this research was obtained from Xianyanglu wastewater treatment plant (Tianjin, China) and stored at 4 °C before use. The inoculums (mesophilic seed sludge) came from an anaerobic reactor, and they were centrifuged before inoculation. Characteristics of the DAS and inoculums were listed in Table 1.

2.2. Pretreatment of the DAS

NaOH and thermal pretreatment were separately performed. The NaOH pretreatment was implemented in a beaker with a working volume of 1.0 L. The DAS was mixed evenly with NaOH with a dose of NaOH of 20, 40, 60 and 80 mg g^{-1} TS DAS, respectively. Then the samples were placed into a refrigerator (4 °C) for 24 h prior to being neutralised to initial pH 7.82 with 6 mol L^{-1} HCl. The thermal pretreatment was performed in a thermal reactor with a working volume of 0.5 L. The DAS was introduced into a thermal reactor at room temperature, and was then heated at 80, 100 and 120 °C for 1 h in autoclave, respectively. After treatment, the samples were put into a refrigerator to guarantee a fast cooling. An untreated sample was also assessed as a reference for the treated samples.

Table 1
Characteristics of DAS and inoculums.

Parameters	DAS	Inoculums
pH	7.82	8.35
TS (%)	22.87	14.94
VS/TS (%)	47.26	69.53
COD (mg kg^{-1})	158312.29	132890.66
SCOD (mg kg^{-1})	1518.02	16694.61
TKN (mg kg^{-1})	9239.20	8325.32
TAN (mg kg^{-1})	431.56	1999.27
VFAs (mg kg^{-1})	182.82	492.36

2.3. Batch experiments of anaerobic digestion

Pressure bottles, with the working volume of 300 ml, were used as digestion reactors. For each treatment run, a mixture of 75 g DAS with 25 g inoculums was used for each reactor, and three parallel reactors were operated. Before anaerobic digestion, oxygen in reactors was removed by exchanging it with nitrogen gas for 5 min. Then reactors were sealed with butyl rubber stoppers. The mesophilic anaerobic digestion was run for 30 days at 37 ± 1 °C to evaluate the effects of pretreatment and high-solids feeding on the methane production. Volumes of produced biogas were based on pressure measurement every day.

2.4. Analytical methods

TS and VS were determined by heating the sludge at 105 °C for 24 h and 550 °C for 4 h, respectively. The samples were first centrifuged at 10,000 rpm for 10 min at 4 °C. And then the supernatant was filtered through a microfiber membrane with a pore size of 0.45 μm to obtain soluble fractions. COD, carbohydrates, total proteins, soluble proteins and TAN were analysed according to dichromate reflux method (CODcr), the Anthrone method, Kjeldahl nitrogen method, Coomassie brilliant blue method, and Nessler's reagent spectrophotometry, respectively (APHA, 1998). Prior to VFAs analysis, the supernatant was acidified by formic acid to adjust the pH to approximately 3.0. Then VFAs were analysed on the Thermal Trace-1300 gas chromatograph equipped with a TR-FFAP column (length 30 m, diameter 0.53 mm) and a flame ionisation detector. The biogas composition was also analysed on a Thermal Trace-1300 gas chromatograph equipped with a Molecular Sieve column (length 2 m, diameter 2 mm) and a thermal conductivity detector.

The degree of solubilisation and biological degradation rate (BDR) of the organic components are calculated, respectively, by the following equations:

$$\text{Degree of solubilisation } (\%) = \frac{\text{soluble concentration}_{\text{treated}} - \text{soluble concentration}_{\text{untreated}}}{\text{total concentration}_{\text{untreated}}} \times 100 \quad (1)$$

$$\text{BDR } (\%) = \frac{\text{total concentration}_{\text{before digestion}} - \text{total concentration}_{\text{after digestion}}}{\text{total concentration}_{\text{before digestion}}} \times 100 \quad (2)$$

3. Results and discussion

3.1. DAS solubilisation after NaOH and thermal pretreatment

3.1.1. COD solubilisation

For NaOH pretreatment (shown in Table 2), the concentration of soluble COD (SCOD) increased with the increase of NaOH dose.