



# Enhancing biological denitrification with adding sludge liquor of hydrolytic acidification pretreated by high-pressure homogenization



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## ARTICLE INFO

### Article history:

Received 30 January 2016

Received in revised form

8 April 2016

Accepted 8 April 2016

Available online 19 April 2016

### Keywords:

High-pressure homogenization

Hydrolytic acidification

Excess activated sludge

Volatile fatty acids

Denitrification carbon source

## ABSTRACT

In this study, high-pressure homogenization (HPH) pretreatment was chosen to enhance the hydrolytic acidification of excess sludge, and the sludge liquor obtained was added to a sequencing batch reactor (SBR) as external carbon source to enhance nitrogen removal. The results indicated that volatile fatty acids (VFAs), sludge disintegration degree ( $DD_{COD}$ ) and  $NH_4^+-N$  increased with increasing the homogenization pressure. The maximum VFA concentration of 1936.0 mg/L was obtained after 3-day hydrolytic acidification with a HPH pretreatment of 40 MPa; acetic acid accounted for 54.4%, propionic acid for 18.3%, and other acids for 27.3% in the total VFAs. The sludge liquor of hydrolytic acidification with HPH disintegration (SLDHA) showed the best performance for wastewater denitrification, compared with the sludge liquor disintegrated alone or hydrolytically acidified alone. When the SLDHA was used as external denitrification carbon source, the total nitrogen removal increased by 51.0% compared to the control.

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## 1. Introduction

In a biological nitrogen removal process, external organic compounds, such as methanol or acetic acid, are usually added to denitrification tank to enhance the total nitrogen (TN) removal. In many wastewater treatment plants (WWTPs), input of these readily degradable organic matters becomes a key step for a successful nitrogen removal (Zhou et al., 2010), thus leads to the increase of operating cost.

The activated sludge process is worldwide used for municipal and industrial wastewater treatment. As a result, a large amount of excess sludge is produced, and needs high treatment and disposal cost, accounting for 25–65% of the total operating cost of WWTPs (Nguyen et al., 2014; Wu et al., 2014). Thus, it is crucial to reasonably reduce and reuse the excess sludge in WWTPs. The excess sludge contains abundant organic substances, like proteins and polysaccharides. These organic substances can be effectively released and degraded to readily biodegradable volatile fatty acids (VFAs) in sludge hydrolysis and acidification process. The generated VFAs have been usually utilized to produce biogas (Guo et al., 2008),

to generate electricity (Jiang et al., 2009), and also to enhance the biological nitrogen removal or simultaneous nitrogen and phosphorus removal as a preferred external carbon source (Tong and Chen, 2009).

However, the efficiency of sludge hydrolysis and acidification is low with less production of VFA if the excess sludge isn't pretreated. The pretreatment disintegrates the excess sludge and enhances the extracellular polymer substance release, which is beneficial to the sludge hydrolysis and acidification. Thus, several efforts have been made to enhance the hydrolytic acidification or anaerobic digestion, such as thermal (Morgan-Sagastume et al., 2011), thermochemical (Vlyssides and Karlis, 2004), mechanical (Fang et al., 2015), electrochemical (Yu et al., 2014), ultrasonic (Kavitha et al., 2015), alkaline-microwave (Qi et al., 2013) and microbial enzyme pretreatment (Yu et al., 2013b).

Recently, high-pressure homogenization (HPH) has been reported as an efficient pretreatment method for sludge disintegration to enhance the further anaerobic digestion (Fang et al., 2014; Onyeche et al., 2003; Rai and Rao, 2009; Wahidunnabi and Eskicioglu, 2014; Zhang et al., 2012a, 2012c). The excess sludge is disintegrated by HPH pretreatment, which is based on disruption of sludge flocs and microbial cells by a combination of large pressure drop, highly focused turbulent eddies and strong shearing force (Zhang et al., 2012c). The HPH treatment has several advantages, such as no chemical changes or denaturing during cell lysis, high

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disruption efficiency and simple operation (Barjenbruch and Koppel, 2003; Onyeche et al., 2003). Zhang (2012b, 2013a, 2013b) established a HPH sludge disintegration model and concluded the main influencing factors and sludge solubilization behavior. Zhang et al. (2012a) pretreated the sewage sludge with HPH and found that the accumulative biogas was effectively enhanced. However, the HPH disintegrated sewage sludge was seldom utilized to produce external carbon source for wastewater treatment through its hydrolytic acidification. Although the HPH pretreatment is energy-intensive as a mechanical treatment, it is more energy-efficient compared with other pretreatment methods, such as ultrasonic and microwave pretreatment (Zhang et al., 2012b).

There were two main objectives of this study: (1) to improve the efficiency of hydrolytic acidification of excess sludge disintegrated by HPH; (2) to clarify the feasibility of sludge liquor as external denitrification carbon source and thus enhance the nitrogen removal in a sequencing batch reactor (SBR). The effect of HPH pressure on the sludge disintegration and further sludge hydrolytic acidification was also investigated. And the sludge liquor, which was prepared by HPH disintegration alone, hydrolytic acidification alone or combination of both, was added into a SBR system to enhance the TN removal.

## 2. Materials and methods

### 2.1. Materials

The excess sludge was taken from a local WWTP in Beijing, China. The sludge was filtered through an 80 mesh sieve so that the particulate matters were taken out, which may damage the high-pressure homogenizer. Then the sludge was concentrated at least 24 h and the supernatant was poured out. The concentrated sludge was stored at 4 °C in a freezer for further experiments. The average characteristics of concentrated sludge were as follows: pH of 7.1, total solids (TS) of 12.7 g/L, volatile solids (VS) of 7.6 g/L, total chemical oxygen demand (TCOD) of 17,954.2 mg/L, SCOD of 356.4 mg/L, VFA of 26.3 mg/L, and  $\text{NH}_4^+\text{-N}$  of 6.2 mg/L.

The artificial wastewater used in the SBR system was composed of  $\text{C}_6\text{H}_{12}\text{O}_6$  of 300 mg/L,  $\text{NH}_4\text{Cl}$  of 192 mg/L,  $\text{NaHCO}_3$  of 588 mg/L,  $\text{KH}_2\text{PO}_4$  of 11 mg/L,  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  of 82 mg/L,  $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$  of 5 mg/L,  $\text{CaCl}_2$  of 33 mg/L and trace element solution of 1 ml/L. The COD/N ratio of artificial wastewater was about 6:1. The trace element solution was prepared according to Syron and Casey (2008).

### 2.2. Methods

#### 2.2.1. Sludge disintegration by HPH

The concentrated excess sludge of 1 L was disintegrated by a high-pressure homogenizer (JJ-30 L/100, Langfang Shengtong Machinery Co., LTD, China) under different homogenization pressures (Zhang et al., 2013a, 2013b). The disintegrated sludge was separated by a centrifugal (H/T 16 MM, Hunan Herexi Instrument & Equipment Co., Ltd, China) at a speed of 10,000 r/min and the supernatant was filtered with a 0.45  $\mu\text{m}$  polyether sulfone membrane to determine the pH, VFA, SCOD and  $\text{NH}_4^+\text{-N}$  of filtrate.

#### 2.2.2. Sludge hydrolytic acidification

The sludge hydrolytic acidification was carried out in conical flasks with a volume of 500 ml. The disintegrated sludge of 500 ml under different homogenization pressures was added into conical flasks, and the control was added with unpretreated sludge of 500 ml. The flasks were flushed with nitrogen gas for 30 s, and sealed with rubber plugs. The preliminary experiments proved that there was no need to add seed sludge for hydrolytic acidification. The conical flasks were then placed into a vapour-bathing constant

temperature vibrator (SHZ-82, Changzhou Guohua Electric Appliance Co., LTD, China) with a temperature of  $35 \pm 1$  °C and a stirring speed of 100 r/min. The sludge pH was uncontrolled. The hydrolytic acidification lasted for 5 d. The sludge liquor of 25 ml was taken from the conical flasks every 24 h. The liquor was separated by the centrifugal at a speed of 10,000 r/min for 10 min and the supernatant was filtered through a 0.45  $\mu\text{m}$  polyether sulfone membrane. The pH, SCOD, VFA and  $\text{NH}_4^+\text{-N}$  of filtrate were analyzed.

#### 2.2.3. SBR sewage treatment system

Two SBR systems were built to treat the artificial wastewater for biological nitrogen removal. The SBR was operated with an aerobic-anoxic model and ran three cycles per day. The operating procedure of SBR was: inflow of 10 min, aeration of 3.5 h, stirring of 3.5 h, sedimentation of 45 min, and drawing of 5 min. The SBR systems were inoculated with activated sludge from a local WWTP in Beijing, China, and cultivated for 3 weeks to ensure the SBR systems reached a stable state, where the effluent COD and TN did not change obviously. Then, three kinds of denitrification carbon sources were added in the anoxic stage to testify their effect for nitrogen removal. Sludge liquor disintegrated (SLD) was produced from raw sludge with HPH disintegration, and the disintegrated sludge mixture was separated by the centrifugal at a speed of 10,000 r/min and the supernatant was filtered through a 0.45  $\mu\text{m}$  polyether sulfone membrane. Sludge liquor hydrolytically acidified (SLHA) was produced from raw sludge by anaerobic hydrolysis and acidification. Sludge liquor disintegrated and hydrolytically acidified (SLDHA) was generated from the HPH disintegrated sludge with anaerobic hydrolysis and acidification. The obtained sludge mixtures were all separated and filtered under the same conditions.

According to calculation, the sludge liquor corresponding to 100 mg/L COD was added to the SBR in the anoxic stage to reach a COD/N ratio of about 8:1. After the aerobic-anoxic wastewater treatment, the effluent was taken out to determine the COD,  $\text{NH}_4^+\text{-N}$ ,  $\text{NO}_2^-\text{-N}$ ,  $\text{NO}_3^-\text{-N}$ , and TN concentration.

### 2.3. Analytical methods

The analyses of sludge TS, VS, TCOD, SCOD and  $\text{NH}_4^+\text{-N}$  were conducted according to standard methods (Eaton et al., 2005). The  $\text{NO}_2^-\text{-N}$  and  $\text{NO}_3^-\text{-N}$  of filtrate and effluent were measured by UV-spectrophotometer (HI 83,200, Hanna Instruments Inc., USA). The TN was calculated as the sum of  $\text{NH}_4^+\text{-N}$ ,  $\text{NO}_2^-\text{-N}$  and  $\text{NO}_3^-\text{-N}$ . The carbohydrate was measured by the phenol-sulfuric method with glucose as the standard (Herbert et al., 1971). The soluble protein was determined by the Lowry-Folin method with BSA as the standard (Lowry et al., 1951).

The efficiency of sludge disintegration was evaluated by disintegration degree ( $DD_{\text{COD}}$ ), which was calculated as Eq. (1) (Bougrier et al., 2005).

$$DD_{\text{COD}}(\%) = \frac{\text{SCOD} - \text{SCOD}_0}{\text{TCOD} - \text{SCOD}_0} \times 100\% \quad (1)$$

where  $\text{SCOD}_0$  is the SCOD of unpretreated sludge.

To analyze the VFAs, the sludge filtrate was further acidified with 3%  $\text{H}_3\text{PO}_4$  to a pH of 4.0, and the acidified liquor of 1.5 ml was collected for gas chromatography test. The VFAs including acetic, propionic, iso-butyric, n-butyric, iso-valeric and n-valeric acid were analyzed by an SP3420A GC (Beijing Beifen-Ruili Analytical Ins. China) with flame ionization detector. The initial temperature of oven was 100 °C for 1 min, the column temperature increased to 210 °C for 3 min with an increase speed of 10 °C/min. Nitrogen gas was used as carrier gas with a flow rate of 30 ml/min. The sample injection volume was 1  $\mu\text{l}$ .

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