



# Stepwise hydrolysis to improve carbon releasing efficiency from sludge



Hongbo Liu <sup>a, b</sup>, Yuanyuan Wang <sup>a</sup>, Ling Wang <sup>a</sup>, Tiantian Yu <sup>a</sup>, Bo Fu <sup>a, b</sup>, He Liu <sup>a, b, \*</sup>

<sup>a</sup> Jiangsu Key Laboratory of Anaerobic Biotechnology, School of Environmental and Civil Engineering, Jiangnan University, Wuxi 214122, PR China

<sup>b</sup> Jiangsu Collaborative Innovation Center of Technology and Material of Water Treatment, Suzhou 215011, PR China

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## ABSTRACT

Based on thermal alkaline hydrolysis (TAH), a novel strategy of stepwise hydrolysis was developed to improve carbon releasing efficiency from waste activated sludge (WAS). By stepwise increasing hydrolysis intensity, conventional sludge hydrolysis (the control) was divided into four stages for separately recovering sludge carbon sources with different bonding strengths, namely stage 1 (60 °C, pH 6.0–8.0), stage 2 (80 °C, pH 6.0–8.0), stage 3 (80 °C, pH 10.0) and stage 4 (90 °C, pH 12.0). Results indicate stepwise hydrolysis could enhance the amount of released soluble chemical oxygen demand (SCOD) for almost 2 times, from 7200 to 14,693 mg/L, and the released carbon presented better biodegradability, with BOD/COD of 0.47 and volatile fatty acids (VFAs) yield of 0.37 g VFAs/g SCOD via anaerobic fermentation. Moreover, stepwise hydrolysis also improved the dewaterability of hydrolyzed sludge, capillary suction time (CST) reducing from 2500 to 1600 s. Economic assessment indicates stepwise hydrolysis shows less alkali demand and lower thermal energy consumption than those of the control. Furthermore, results of this study help support the concepts of improving carbon recovery in wastewater by manipulating WAS composition and the idea of classifiably recovering the nutrients in WAS.

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## 1. Introduction

In recent years, WAS yield has increased year by year in wastewater plants (WWTPs) of China, and reached 55 million tons with water content of 80% in 2014 (National Bureau of Statistics of China, 2015). WAS disposal has become a critical issue. Recycling carbon source from WAS has gradually been a hot research (Kim et al., 2009; Yan et al., 2013; Liu et al., 2012), and physic-chemical hydrolysis of WAS pretreatment is even listed as one of the 18 most promising technologies in the field of wastewater treatment in future by the International Water Association (IWA) (2016).

Recent development of sludge hydrolysis could be generally divided into two trends: high-strength hydrolysis and mild hydrolysis. The former is often used to direct recovery sludge carbon source, in the process of which, protein and polysaccharide could be directly hydrolyzed into small molecular substances, such as

amino acids and simple sugars. For example, Su et al. (2014) obtained amino acids from municipal excess sludge by thermal hydrolysis and used it as the inhibitor for steel corrosion. However, the high energy consumption and harsh equipment requirements seriously hampered its widely application (Koottatep et al., 2016). Mild hydrolysis with low energy consumption and equipment requirements is still widely adopted in the field of sludge pretreatment for enhancing the yields of VFAs or methane in sludge anaerobic fermentation processes (Pei et al., 2016; Yao et al., 2016). There are many kinds of sludge pretreatment methods reported in previous studies, including mechanical, chemical and biological methods, amongst of which, TAH is considered as one of the most effective methods (Hyun et al., 2013; Kim et al., 2013) and was thus implemented for sludge hydrolysis in this test. However, there are also some shortages in mild hydrolysis, such as inadequate release of carbon resource, low biodegradability of the released SCOD, poor dewaterability of hydrolyzed sludge and low recovery of released carbon (Barber, 2016; Tong and Chen, 2009; Xiao et al., 2016).

Carbon source in sludge is mainly composed of organics absorbed on the surface of sludge flocs (OM), loosely bound extracellular polymeric substances (LB-EPS), tightly bound extracellular

\* Corresponding author. Jiangsu Key Laboratory of Anaerobic Biotechnology, School of Environmental and Civil Engineering, Jiangnan University, Wuxi 214122, PR China.

E-mail address: [liuhe@jiangnan.edu.cn](mailto:liuhe@jiangnan.edu.cn) (H. Liu).

polymeric substances (TB-EPS) and the intracellular matters (IM), which are entrapped in the matrix of sludge flocs at different bonding strengths. In conventional hydrolysis process, uniform hydrolysis strength and reacting time are evenly exposed on those organics distributing at different parts of sludge. Therefore, during mild hydrolysis, those organics with high bonding strengths, such as intracellular matters, could not be complete released, resulting in the low SCOD yields, while other organics with low bonding strengths, mainly extracellular organics, could be easily released and then further reacts to produce non-biodegradable substances. For example, some non-biodegradable matters, such as melanin and melanoidin, could be produced in the process of alkaline thermal hydrolysis by Maillard reactions, or called carbonyl amino reactions, between carbonyl compounds (reducing sugars) and amino compounds (amino acids and proteins), which resulted in the low biodegradability of the released carbon (Neyens et al., 2003; Salsabil et al., 2010). Moreover, during the conventional mild hydrolysis, abundant of dissolved organics would be released and accumulated in the hydrolyzed sludge system. Previous researches indicated the existence of superabundant soluble organics, especially protein and polysaccharide, in the hydrolyzed sludge was the reason to its poor dewaterability (Zhu et al., 2015).

Therefore, based on the distribution and bonding strength of organics in WAS, a novel strategy, stepwise release of carbon source from sludge, was developed in this research to systematically solve those problems mentioned above in mild hydrolysis. By stepwise improving hydrolysis strength and timely recovering sludge carbon in a planned way, (1) the hydrolysis force is directly exposed on the targeted organics in sludge, which could greatly improve hydrolysis efficiency and enhance the amount of released sludge carbon, (2) the released carbon avoids to be excessively hydrolyzed, which could improve the quality of the released carbon, and (3) the accumulations of protein and polysaccharide in hydrolyzed sludge system can be eased, which could alleviate the deterioration of sludge dewaterability. The objective of this study is to provide a mild hydrolysis technology for efficiently recovering sludge carbon. The amount and quality of the released carbon were investigated, and efficiency in improving sludge dewaterability were analyzed. By exploiting the mechanisms of stepwise hydrolysis, the concept of improving carbon recovery in wastewater by manipulating WAS composition and the feasibility of classifiably recovering the nutrients in WAS were discussed.

## 2. Materials and methods

### 2.1. Substrates

WAS used as the substrate for hydrolysis was taken from the sludge storage tank of a local WWTP in Wuxi city, China. An intermittent cycle extended aeration/membrane bioreactor process is used in this WWTP. The fresh WAS was pre-concentrated before used. The concentrated WAS had pH of 6.5–7.5, total suspended solids (TSS) concentration of 30.0–31.0 g/L, volatile suspended solids (VSS) concentration of 15.0–16.0 g/L, CST of 40.0–50.0 s, average volume particle diameter of 70.0–80.0  $\mu\text{m}$ , SCOD of 130.0–140.0 mg/L, soluble protein of 13.0–15.0 mg/L and soluble polysaccharides of 45.0–50.0 mg/L. All measurements were conducted in triplicate.

### 2.2. Seeding sludge for anaerobic fermentation

Anaerobic sludge from an up-flow anaerobic sludge blanket (UASB) for brewery wastewater treatment was collected as the seeding sludge. In order to accumulate acetogenic bacteria, the anaerobic sludge was firstly concentrated by setting for 24.0 h at

ambient temperature and the precipitated sludge was then treated at 105 °C for 2.0 h to kill methanogens. To reactivate acetogenic bacteria, the heat-treated sludge was added into a 1000 ml shaking flask holding nutrient solution whose compositions, concluding 3.0 g/L glucose, were referred to previous reports (Jiunn et al., 2003; Ginkel and Logan, 2005). When the mixed-liquor suspended solids (MLSS) was 9.0 g/L, pH was about 6.5, stirring speed was 120.0 rpm and temperature was 35.0 °C, the heat-treated sludge was cultivated for 24.0 h in the completely anaerobic flask. Finally, the seeding sludge was obtained by centrifuging the cultivated sludge at 4800 rpm for 10 min. In cultivation process, pH was adjusted by dilute HCl and NaOH, oxygen in headspace of flask was removed by injecting nitrogen for 10.0 min, dissolved oxygen was removed by adding L-cysteine solution and phosphate was used as the buffer.

### 2.3. Strategy for WAS stepwise hydrolysis

As shown in Fig. 1, the strategy of WAS stepwise hydrolysis could be divided into 4 stages. In stage 1, the conditions were controlled at temperature of 60 °C and pH of 6.0–8.0. WAS with TSS concentration of about 30.0 g/L was continuously stirred for 3.0 h, aiming to strip the carbon source absorbed on the surface of sludge. Then, the hydrolyzed WAS was centrifuged to separate the liquor and solid. The former contains the carbon source released in stage 1#. The solid fraction was adjusted to the initial TSS concentration of about 30.0 g/L by adding tap water and used for subsequent hydrolysis in stage 2, where the conditions were controlled at temperature of 80 °C and pH of 6.0–8.0. In this stage, the WAS, that is, the solid fraction of stage 1, was also continuously stirred for 3.0 h in order to strip the sludge carbon distributing outside of sludge cell. Then, the liquor and solid in the hydrolyzed sludge was separated by centrifugation. The liquor fraction contains the carbon source released in stage 2#. The solid fraction was adjusted to the initial TSS concentration of about 30.0 g/L by adding tap water for subsequent hydrolysis. Under the same processes, the residual sludge in stage 2 was further hydrolyzed during stages 3 and 4 in sequence. The conditions in stage 3 were controlled at temperature of 80 °C, pH of 10 and stirring time of 6.0 h; and in stage 4 were temperature of 90 °C, pH of 12 and stirring time of 6.0 h. In all stages, the centrifugal intensity was controlled at  $7000 \times g$  for 10 min. The liquor fractions were collected separately, and then anaerobic fermented for acids production, respectively. Moreover, as shown in Fig. 1, the conventional TAH was implemented as the control, in which, the conditions were controlled at temperature of 90 °C, pH of 12 and stirring time of 3.0 h.

### 2.4. Anaerobic fermentation for VFAs production

Six beaker flasks of 500 ml with equal amount of seeding sludge were filled with 250 ml supernatant of the hydrolyzed WAS in stage 1 (L1), stage 2 (L2), stage 3 (L3), stage 4 (L4) and the control (CL), respectively, and adjusted pH of 10.0 by dilute HCl and NaOH. Dissolved oxygen in supernatant and gaseous in the headspace of flasks were removed by sparging gaseous nitrogen for about 30 min to maintain strict anaerobic condition. In the whole process of fermentation, flasks were placed in orbital shaker with rotation speed of 120 rpm, temperature was kept at about 35 °C and pH was kept at 10.0. Samples taken from beaker flasks at certain intervals were analyzed. All the experiments were carried out independently in triplicates.

### 2.5. Analytical methods

Samples were pretreated by filtering with GF/C glass microfiber of 0.45  $\mu\text{m}$ . Conventional indexes, including COD, BOD, VSS, TSS and

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