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Phosphorus and short-chain fatty acids recovery from waste activated sludge by anaerobic fermentation: Effect of acid or alkali pretreatment

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HIGHLIGHTS

- P and SCFAs recovery from WAS by AF with acid or alkali pretreatment was proposed.
- Both acid and alkali pretreatment effectively enhanced SCFAs production by AF.
- Alkali pretreatment enhanced conversion of organic P to inorganic P.
- Acid pretreatment was more beneficial for inorganic P release into solution.
- Acid pretreatment is preferred for simultaneous recovery of P and SCFAs by AF.

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ABSTRACT

Waste activated sludge (WAS) was pretreated by acid or alkali to enhance the anaerobic fermentation (AF) for phosphorus (P) and short-chain fatty acids (SCFAs) release into the liquid simultaneously. With acid pretreatment, the released total P concentration achieved 120 mg/L, which was 71.4% higher than that with alkali pretreatment. In addition, alkali pretreatment enhanced organic P release with about 35.3% of organic P in the solid being converted to inorganic P, while little had changed with acid pretreatment. The results also showed that acid and alkali pretreatment enhanced SCFAs production by 15.3 and 12.5 times, respectively. Acid pretreatment could be preferred for simultaneous recovery of P and SCFAs by AF.

1. Introduction

Phosphorus (P) is one of the most important nutrients to all life (Cooper et al., 2011; Cordell et al., 2009; Rittmann et al., 2011). However, P rock is non-renewable, which has been reported that P supply is less than the demand after 2035(Cordell et al., 2009). So new source of P need to be found as soon as possible. Because of the difference of P concentration in wastewater, the used P removal process and its efficiency, waste activated sludge (WAS) contains1.5–3.0% phosphorus (P) as the authors investigated a number of WAS samples from different municipal wastewater treatment plants in Shanghai. Furthermore, WAS from enhanced biological phosphorus removal (EBPR) process in wastewater treatment plants (WWTPs), with P content of 5–8%, could be an important source for P recovery (Garcia Martin et al., 2006; Xie et al., 2011). Thus, studies on P recovery from WAS have been intensively carried out.

Wang et al. used conventional electrodialysis for P recovery from WAS, and the recovery ratio of P in the solution could be 95.8% (Wang et al., 2013). In Chen's study, P was recovered from alkaline pretreated WAS with fermentation, and crystallized as struvite, with 92.8% of the soluble orthophosphate (PO_4^3-$P_{(L)}$) being recovered (Tong and Chen, 2007). However, these high P recovery efficiencies were based upon PO_4^3-$P_{(L)}$ amount released from WAS in the solution. If based on the total P (TP) in WAS, the calculated P recovery efficiencies were only about 40–50% (Nguyen et al., 2014). Therefore, the release of P from the solid phase to the liquid phase is the key issue for P recovery from WAS (Xu et al., 2015).

Anaerobic fermentation (AF), as an environmentally friendly process without much chemical agent needed, has been largely studied for WAS treatment (Huang et al., 2015; Sun et al., 2014). It's an effective treatment process to reduce WAS amount as well as produce/recover energy (Appels et al., 2008). During AF, most of the P stored as polyphosphates and part of the P in the organic matter could release into liquid solution (Pastor et al., 2008). In

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addition, it was favorable for P release when anaerobic fermentation process was controlled at the acidogenic stage (He et al., 2016).

At the same time, the short-chain fatty acids (SCFAs) produced by AF can be recovered as a promising carbon source for denitrification in the wastewater treatment process (Zhang and Chen, 2009), as SCFAs are the preferred carbon sources for biological nutrient removal microbes (Jiang et al., 2007). Thus, AF of WAS could be a great way for both P and SCFA recovery.

As regards the direct AF of WAS by microorganisms, its distinctive structure makes it difficult and time-consuming (Huang et al., 2015). Therefore, pretreatment is always needed to enhance AF efficiency and further improve P release efficiency (Cui and Shen, 2012). Recently, various pretreatment technologies have been developed to promote AF of WAS (Carrere et al., 2010; Yuan et al., 2006), such as mechanical (Yang et al., 2015), chemical (Cui and Shen, 2012; He et al., 2016) and combined pretreatment methods (Wang et al., 2016). Among these methods, acid or alkali pretreatment has good application prospect due to its simplicity, no special equipment's required, and less time consuming. Moreover, acid or alkali pretreatment also does good to improve inorganic P (IP) release. But in these studies, the researches looked P release as a spontaneous process with only the final released P concentration being investigated. In fact, there are three P species in WAS, including organic P (OP), apatite P (AP) and non-apatite inorganic P (NAIP) according to the Standards Measurements and Testing (SMT), and the fraction of inorganic P always account for a great part (Xie et al., 2011). The release performance of these three P species depend greatly on the WAS pretreatment and the anaerobic fermentation condition. The pH value would affect the release of inorganic P greatly, possibly with different trend to that of SCFAs release during AF. In our latest study on P release from WAS, it was made clear that the release of IP was improved under acid condition, and the release of both NAIP and OP was improved under alkaline condition (Xu et al., 2015). Thus the authors were motivated to investigate how the acid and alkali pretreatment could improve the P and SCFA recovery from WAS simultaneously.

The main objectives of this study were to investigate the effects of acid or alkali pretreatment on P release and P speciation conversion during AF, as well as on SCFAs production enhancement. It is hoped that the pretreatment methods would facilitate P release and enhance SCFAs yield, being recovered simultaneously from WAS.

2. Materials and methods

2.1. WAS and inoculated sludge

The WAS sample used in this research was collected from a municipal WWTP in Shanghai, China, and preserved at 4 °C. WAS is rich in P with 2%.

Anaerobic granular sludge was used as inoculated sludge (IS) in this work, which was obtained from a landfill plant in Shanghai, China.

The characteristics of WAS, IS and WAS + IS are listed in Table 1.

2.2. Sludge pretreatment

To enhance anaerobic P releasing, the WAS was pretreated using acid and alkaline method before AF. In the pretreatment, 6 M hydrochloric (HCl) or 6 M sodium hydroxide (NaOH) was added to the 2 L of WAS for pH adjustment to 3.0 or 10.0. Then the samples were mixed on a magnetic stirrer at 250 rpm for 30 min. After pretreatment, the sludge was used for batch AF.

Table 1
Characteristics of SS, IS and IS + SS before the AF.

Parameter	Unit	WAS	IS	WAS + IS
pH	–	6.7	7.8	7.9
TS	g/L	14.7	60.1	22.3
VS	g/L	10.3	30.4	13.7
TCOD	g/L	3.5	36.9	9.8
SCOD	mg/L	160	4960	960
$TP_{(L)}$	mg/L	58.4	17.8	51.6
PO_4^{3-}-$P_{(L)}$	mg/L	31.5	11.7	28.2
NH_4^+-N	mg/L	71.7	374.9	122.2
Soluble protein	mg/L	104.8	75.2	99.9
Soluble Polysaccharide	mg/L	37.9	29.8	36.5

2.3. Batch fermentation

The batch experiment set-up were carried out in 150 mL headspace bottles coupled with 500 mL gas-collecting pockets. The 100 mL pretreated WAS and 20 mL IS were mixed and then transferred to the headspace bottles for the AF. Meanwhile, WAS without pretreated was set as the blank test. Before AF, the bottles were purged with nitrogen gas for 3 min to maintain anaerobic conditions. Then 2 M 2-bromoethanesulphonate (3 mL) was added to inhibit methane production. All the bottles were placed into a water-bathing with vibrator speed of 160 ± 10 rpm at 35 ± 1 °C. The duration of the fermentation was 84 h. Totally 54 bottles were used for the three groups (acid pretreated, alkali pretreated and untreated), with every 18 bottles for each group, respectively. For each group, two (as duplicates) of the 18 bottles were taken away for sample analysis every 12 h, as the data of the corresponding time points (nine points totally). Samples were immediately centrifuged at 3000 rpm for 10 min and then the supernatant was filtered with a 0.45 μm cellulose membrane for total P ($TP_{(L)}$) and PO_4^{3-}-P analyses. The sludge residues were dried at 105 °C for 24 h to constant weight, and grinded in a mortar to obtain a fine powder for analyzing P fractions by SMT (Xie et al., 2011). Each test was performed with two duplicates.

2.4. Analytical methods

Scanning electron microscopy (SEM) analysis was performed for surface morphology investigation of the WAS with and without pretreatment using a JSM-6700F instrument (JEOL Co., Ltd., Japan). The pH was monitored by a pH meter (PHS-3C) with a combined electrode (E-201-C). The analyses of PO_4^{3-}-P, $TP_{(L)}$, total nitrogen (TN), NH_4^+-N and solubility of chemical oxygen demand (SCOD) were performed following standard methods (APHA, 2012). Organophosphorus in the liquid ($OP_{(L)}$) was calculated as the difference between $TP_{(L)}$ and PO_4^{3-}-P (Juston and DeBusk, 2011).

SCFAs analyses were performed using Agilent 7890 N GC equipped with flame ionization detector (FID) and DB-WAX capillary column (30 m × 530 μm × l μm). The flow rate of the carrier gas (N_2) was 50 mL/min. The injection and detector temperatures were 220 °C and 250 °C, respectively. The oven began at 110 °C and remained for 2 min, then increased at a rate of 10 °C/min to 200 °C, and hold at 200 °C for 2 min. The sample injection volume was 1.0 μL. Chemical oxygen demand (COD) conversion factors are 1.1 g COD/g acetic acid (HAc), 1.5 g COD/g propionic acid (HPr), 1.8 g COD/g butyric acid (HBu), 2.0 g COD/g valeric acid (HVa).

The P fractions in the solid were classified as total phosphorus ($TP_{(S)}$), IP, NAIP, AP and organophosphorus ($OP_{(S)}$) by the SMT protocol (Xie et al., 2011). That is $TP_{(S)}$ = IP + $OP_{(S)}$ and IP = NAIP + AP. In all cases, different P fractions were determined in the extracts according to the molybdenum blue method as described above.