



Alkaline solubilization of excess mixed sludge and the recovery of released phosphorus as magnesium ammonium phosphate

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ARTICLE INFO

Keywords:

Excess mixed sludge
Alkaline solubilization
Phosphorus forms
Phosphorus recovery

ABSTRACT

The alkaline solubilization of excess mixed sludge was investigated and subsequently the released phosphorus was recovered as magnesium ammonium phosphate (MAP). Considerable and rapid release of glycogen and protein was encountered after alkaline addition into the sludge. Only 45.0% of sludge cells were destructed after 240 min treatment in R1 while the corresponding ratio was 96.1% in R3 according to the release of DNA. Non-apatite inorganic phosphorus (NAIP) in the alkaline treated sludge decreased due to the dissolution of $\text{Al}(\text{OH})_3$ and AlPO_4 , which was the dominant reason for phosphorus release. Soluble orthophosphate (SOP) in the supernatant of alkaline treated sludge could be recovered quickly with the recovery efficiency of 84.6% within 5 min and about 53–55% of P participated in MAP reaction, producing large amounts of acicular crystals.

1. Introduction

Phosphorus (P) is an irreplaceable elemental resource, which is critical for the sustainability of human beings, and it is estimated that phosphate rock reserves may be exhausted in 60–80 years (Hong et al., 2005; Rittmann et al., 2011). Consequently, it is increasingly important to develop technologies to recover P from various waste streams, thus contributing to reducing the consumption of phosphate rock.

Excessive P discharge causes eutrophication in effluent receiving surface waters, therefore various biological and chemical methods have been widely applied in wastewater treatment in order to meet the stringent P regulation in the wastewater effluent. Enhanced biological P removal (EBPR) is the most popular biological approach through which P is enriched in the biomass cells discharged as the excessive activated sludge. On the other hand, $\text{Fe}^{2+}/\text{Fe}^{3+}$ or Al^{3+} based salts such as $\text{FeCl}_2/\text{FeCl}_3$, $\text{Al}_2(\text{SO}_4)_3$, polyaluminum chlorides (PAC) are widely used for chemical P removal, producing amounts of chemical sludge. Compared to other P removal reagents, PAC produces larger and stronger flocs than alum and provides more space for adsorption (Gregory and Dupon, 2001), and can remove phosphates and particulate matters by the mechanism of precipitation and adsorption because of ionic state of Al in PAC (Kim et al., 2002), thus PAC has been used extensively for P removal from wastewater. During wastewater treatment with chemical and/or EBPR around 90% of the P-load in the raw

sewage (1.8 g P/(capita d)) is removed from the wastewater and transferred into the sewage sludge (Petzet et al., 2012), hence the disposal of P rich sludge is a P loss when considering the inadequate P resource, and P recovery needs to be proceeded.

In terms of wastewater sludge, the sources for P release and recovery mainly include sewage sludge, digester supernatant and sewage sludge ash (SSA). Sewage sludge is the residue resulting from the wastewater treatment process, and wet-chemical/physical approaches are required to disintegrate sludge and release P with significant sludge reduction followed by P recovery. Various processes have been proposed, such as Seaborne approach, Stuttgart approach and Budenheim process, which were summarized by Egle et al. (2015). The digester supernatant is the liquid flow from the thickening and/or dewatering of digested sludge, and P recovery is easily carried out since significant P is released in the flow as $\text{PO}_4^{3-}\text{-P}$ through sludge digestion. Magnesium ammonium phosphate hexahydrate (MAP) method was applied to recover P and nitrogen from the supernatant of anaerobic digestion sludge and alkaline fermentation liquid of excess sludge (Bi et al., 2014). Tong and Chen (2007) concluded that 92.8% of the soluble orthophosphate (SOP) could be recovered from the alkaline fermentation liquid of waste activated sludge with Mg/P (mol/mol) = 1.8, pH 10 and 2 min reaction time. So far, many literatures focus on P release and recovery from SSA (Petzet et al., 2012; Takahashi et al., 2001; Adam et al., 2009; Petzet et al., 2011), and acid and alkali addition are

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the dominant techniques for P dissolution. Acid input such as H_2SO_4 (Franz, 2008), HNO_3 (Sano et al., 2012) can leach Al and Ca combined P (Al-P, Ca-P) but also dissolve heavy metals, which are undesirable when recovered P is used as fertilizer. In contrast, alkaline cannot extract Ca-P and heavy metals but dissolve Al-P and Fe bound P (Fe-P).

In many wastewater treatment plants (WWTPs), EBPR is not competent to meeting the stringent standard of effluent P and thus such P removal agents as PAC, $FeCl_2$ are added into the aeration tank to assist in P removal. In this case, the discharged waste sludge is divided into two categories, i.e. the mixture of excessive activated sludge and chemical sludge, both of which contain abundant P. The former undergoes the EBPR process assimilating much P and the latter immobilizes considerable P via chemical precipitation and adsorption. Nevertheless, there are few literatures focusing on P release and recovery from this mixed sludge. Ali and Kim (2016) reported P release from PAC treated sludge, which, however, was not the raw sludge but dried and pulverized with mortar to a mesh size < 0.5 mm before acid and alkali leaching, and P recovery investigation was not performed. Bi et al. (2014) recovered P from the alkaline hydrolysis supernatant of excess sludge, which did not contain chemical sludge. Additionally, Tong and Chen (2007) performed P recovery from the alkaline fermentation liquid of waste activated sludge. So far, however, researchers pay no attention to disintegration of this mixed sludge treated with wet-chemical methods, for example alkali addition. It is crucial to understand whether P can release substantially from the mixed sludge since alkaline pretreatment is a commonly examined pretreatment method which is very effective in terms of solubilizing extracellular polymers (Doğan and Sanin, 2009), and can also dissolve Al-P and Fe-P which are formed via chemical P removal. More importantly, the recovery potential of the thus released P should be investigated. In addition, most of the previous literatures focused on P release from sludge but pay no attention to the distribution and transformation of P forms in the treated sludge along with P release, and the discussion about the process of P release was ignored. Consequently, better understanding of the mechanism responsible for P release is needed to optimize treating conditions in future in consideration of the abundant P immobilized in the mixed sludge.

In this study the mixed sludge was treated with alkali and the investigation aims at dealing with issues as follows: (1) sludge disintegration analyzed with the glycogen, protein and deoxyribonucleic acid (DNA) release; (2) P release and the related mechanism analysis in combination with the distribution and transformation of P forms in the sludge; (3) the potential of P recovery as MAP.

2. Material and methods

2.1. Excess mixed sludge

The excess mixed sludge was taken from sludge thickener of a municipal wastewater treatment plant (WWTP) running by University of Capetown (UCT) process in Shenzhen, China. Due to the stringent P discharge management and unsatisfactory P removal efficiency, PAC was added into the aeration tank of the process to enhance P removal. Thus, the discharged sludge was the mixture of excess activated sludge and chemical sludge.

2.2. Experimental design

The experiment consisted of the alkaline solubilization of sludge and the subsequent P recovery. The single factor experiment was performed with 500 mL of excess mixed sludge placed into three 1000 mL beakers (R1, R2 and R3) and simultaneously the sludge was stirred continuously at 200 rpm with magnetic stirrer. Then 12.5 mol/L NaOH was added into the sludge until the pH of sludge was adjusted to 11.0, 12.0 and 13.0 in R1, R2 and R3, respectively. The stirring went on after alkaline addition to promote sludge solubilization, but alkali was not

Table 1
Experimental design of alkaline solubilization of sludge.

Reactor	Initial pH after alkaline addition	Alkaline dosage (mg-NaOH/g-TS)
R1	11.0	69.7
R2	12.0	102.3
R3	13.0	173.4

added again during solubilization to keep the pH stable. The experimental design of alkaline solubilization of sludge was given in Table 1.

The supernatant of the mixed sludge after 240 min alkaline treatment was collected for P recovery as MAP to assess the potential of P recovery. Herein, 500 mL supernatant was input into 1000 mL beaker, and pH = 9.5 was arranged via 9.2 mol/L sulphuric acid addition. In addition, Mg^{2+} and NH_4^+-N was supplied with the molar ratio of Mg/P (mol/mol) = 1.5 and N/P (mol/mol) = 1.5, which was enough for P crystallization as MAP since the molar ratio of Mg, N and P comprising MAP is 1:1:1. Also, stirring was run throughout the P recovery with magnetic stirrer at 200 rpm. Both of the alkaline solubilization and P recovery experiments were carried out in triplicate and the average values were used for the discussion.

2.3. Analysis

During alkaline solubilization of sludge in the reactors, 8 mL sludge sample each time (0 min, 10 min, 30 min, 60 min, 120 min, 240 min) was taken out from the reactors and centrifuged at 2000 rpm for 5 min, and then the supernatant was filtered through a 0.45 μ m-pore-size syringe filter unit before determination. Total phosphorus (TP) and SOP ($PO_3^{2-}-P$) were determined with ammonium molybdate spectrophotometric method (MEP, 2002). Ammonia nitrogen (NH_4^+-N) was determined by salicylic acid spectrophotometry (MEP, 2002). Total nitrogen (TN) was measured by Total nitrogen analyzer (Jena Multi N/C 3100). Chemical oxygen demand (COD) was analyzed via fast digestion – spectrophotometric method (MEP, 2002). Glycogen was measured by phenol – sulfuric acid method and protein was detected by Bradford method (Yu et al., 2017). DNA was analyzed by diphenylamine colorimetric method (Xiao et al., 2015). The metal elements were determined by inductively coupled plasma-optical emission spectrometer (ICP, PerkinElmer NeION 300). The P forms determination was based on the Standard Measurement and Testing (SMT) method (Ruban et al., 1999). Non-apatite inorganic phosphorus (NAIP) is associated with oxides and hydroxides of Fe, Al or Mn (Fe/Al-P), apatite phosphorus (AP) associated with Ca (Ca-P) (Ali and Kim, 2016). The morphological character of precipitate from P recovery was observed by optical microscope (Olympus CX31) equipped with a camera. Furthermore, the precipitate was also examined by X-ray diffraction (XRD) (Germany Bruker D8 Advance).

Many researchers used the released SCOD (soluble chemical oxygen demand) and the corresponding DD (disintegration degree) calculated according to SCOD to evaluate the disintegration degree of sludge pretreated by wet-chemical/physical approaches (Kim et al., 2010; Park et al., 2012). Actually, the SCOD release was derived from the damage of both EPS and microbial cells, therefore DD cannot reflect the disruption of microbial cells. 70–80% of EPS consists of saccharides and protein but less nucleic acid (Houghton and Stephenson, 2002). After the cell walls and cell membranes were damaged, the cell nuclei were also damaged, and DNA was released and converted into soluble matter (Celis, 2005), therefore the released DNA is more appropriate to assess the disruption of microbial cells in sludge exposed to alkaline condition. Xiao et al. (2015) conducted sludge destruction at pH 12.5 and found that the soluble DNA only increased by 10.6% from 5 min to 24 h of alkaline pretreatment, thus Xiao et al. (2015) hypothesized that the microbial cells were cracked completely and regarded the soluble DNA release amount after 24 h alkaline pretreatment as the entire release

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